

ROADS

RECEIVED

FEB 8 1993

6T-EA

Copper Cove Village Asbestos Site  
Copperopolis, CA

Prepared for:

U.S. Environmental Protection Agency  
Region IX  
Emergency Response Section

William E. Lewis  
Deputy Project Officer

Contract Number 68-01-6669

TDD #098604-20  
PCS #5137

Prepared by:

Roy F. Weston, Inc.  
In Association with Tetra Tech, Inc.  
Technical Assistance Team

Date:

July 24, 1986

000044



705354



Suite 420, 120 Howard Street, San Francisco, CA 94105 • (415) 543-5099

TECHNICAL ASSISTANCE TEAM FOR EMERGENCY RESPONSE REMOVAL AND PREVENTION  
EPA CONTRACT 68-01-6669

July 24, 1986

U.S. Environmental Protection Agency  
215 Fremont Street  
San Francisco, CA 94105

PCS #5137  
TDD #098604-20  
TAT #098607-F-0014

Attention: William E. Lewis, DPO

Subject: Copper Cove Village, Copperopolis, CA

Gentlemen:

Enclosed is the report of the asbestos dust mitigation action taken in Copper Cove Village, Copperopolis, California.

If you have any questions regarding this summary report, please do not hesitate to contact this office.

Sincerely,

John King  
Technical Assistance Team Leader

Gary Floyd  
Technical Assistance Team Member

Enclosure

cc: NPMO  
File

Roy F. Weston, Inc.  
SPILL PREVENTION & EMERGENCY RESPONSE DIVISION  
In Association with ICF Inc., Jacobs Engineering Group Inc., C.C. Johnson & Associates, Inc., and Tetra Tech, Inc.,

000045

## Table of Contents

	Page No.
1.0 Site Background . . . . .	1
1.1 Origin of the Asbestos Problem . . . . .	1
1.2 Initial EPA Involvement and Preliminary Assessment . . . . .	1
2.0 Sampling. . . . .	1
2.1 Preliminary Assessment Strategy. . . . .	1
2.2 Removal Action Strategy. . . . .	3
2.3 Sampling Technique . . . . .	3
2.4 Field Sampling Results . . . . .	7
3.0 Laboratory Procedures . . . . .	8
3.1 Analytical Techniques. . . . .	8
3.1.1 Soil Samples. . . . .	8
3.1.2 Air Samples . . . . .	8
3.2 Rationale for Selected Analytical Techniques . . . . .	8
3.3 Quality Assurance/Quality Control. . . . .	12
4.0 Disposal Alternatives for Asbestos. . . . .	12
5.0 Immediate Removal Action. . . . .	12
5.1 Decision to Apply Chip Seal. . . . .	13
5.2 Chip Seal Application. . . . .	13
5.2.1 Phase I (April 30 - May 12, 1986) . . . . .	13
5.2.2 Phase II (May 20 - 22, 1986). . . . .	13
5.3 Air Monitoring . . . . .	16
5.3.1 Test Scenario . . . . .	16
5.4 Weather Monitoring . . . . .	21
5.5 Cost Monitoring. . . . .	21
6.0 Remedial Investigation. . . . .	21
7.0 Summary . . . . .	28
Bibliography. . . . .	29
Appendix I - Photographic Documentation . . . . .	30
Appendix II - Analytical Techniques for Asbestos. . . . .	40
Appendix III - Preliminary Assessment Report. . . . .	74
Appendix IV - Laboratory Analysis Reports . . . . .	77

000046

## List of Figures

Figure No.	Page No.
1 - Site Location Map . . . . .	2
2 - Preliminary Assessment Soil Sample Locations Map . . . . .	4
3 - Removal Action Soil Sample Locations Map. . . . .	5
4 - Copperopolis Elementary School Soil Sample Locations Map . . . . .	6
5 - Chip Sealed Roads in Copper Cove Village. . . . .	15
6 - Test Scenario Site Map. . . . .	20

000047



## List of Tables

Table No.	Page No.
1 - Soil Sampling Data Summary. . . . .	9-11
2 - Comparison of Dust Control Alternatives . . . . .	14
3 - Air Sampling Data Summary . . . . .	17-19
4 - Test Scenario Air Sampling Data Summary . . . . .	22
5 - Weather Monitoring Data . . . . .	23-26
6 - Weather Data Graphic Representation . . . . .	27

00CC48

## **1.0 Site Background**

### **1.1 Origin of the Asbestos Problem**

Copper Cove Village is located approximately 4 miles southeast of Copperopolis, California off of O'Byrnes Ferry Road in Calaveras County, and is depicted on the site location map (Figure 1). The jurisdictional boundaries of this subdivision lie within the Rocky Road Maintenance District.

Construction of Copper Cove Village commenced in the mid-1960's and at that time the aggregate used as roadbed material consisted of crushed serpentine rock. The rock was excavated from area quarries which are located in serpentine rock deposits characteristically known to contain chrysotile. A majority of these roads ( 16 mi.) constructed in Copper Cove Village remained unpaved following completion. Studies in recent years have concluded that airborne asbestos concentrations increase near unpaved roads constructed of crushed serpentine rock due to a number of factors including vehicle traffic, silt content of the road surface, and decreased soil moisture.

### **1.2 Initial EPA Involvement and Preliminary Assessment**

Copperopolis Community Board member Ron Shields, knowing of the health effects of chrysotile fiber inhalation, namely asbestosis, mesothelioma, and lung cancer, informed the Environmental Protection Agency (EPA) Emergency Response Section (ERS) of his concern that dust emitted from the gravel roads within the subdivision may contain asbestos fibers. This notification constituted the initial involvement of EPA with the Copperopolis site.

On November 18, 1985, Technical Assistance Team (TAT) member Steve Wolfe traveled to Copperopolis and met with Ron Shields to discuss the situation. Mr. Shields informed Mr. Wolfe in detail of the potential problem existing in Copper Cove Village in conjunction with the existing unpaved serpentine gravel roads. Mr. Wolfe then conducted a preliminary assessment which included an effective sampling regime and a representative photographic documentation.

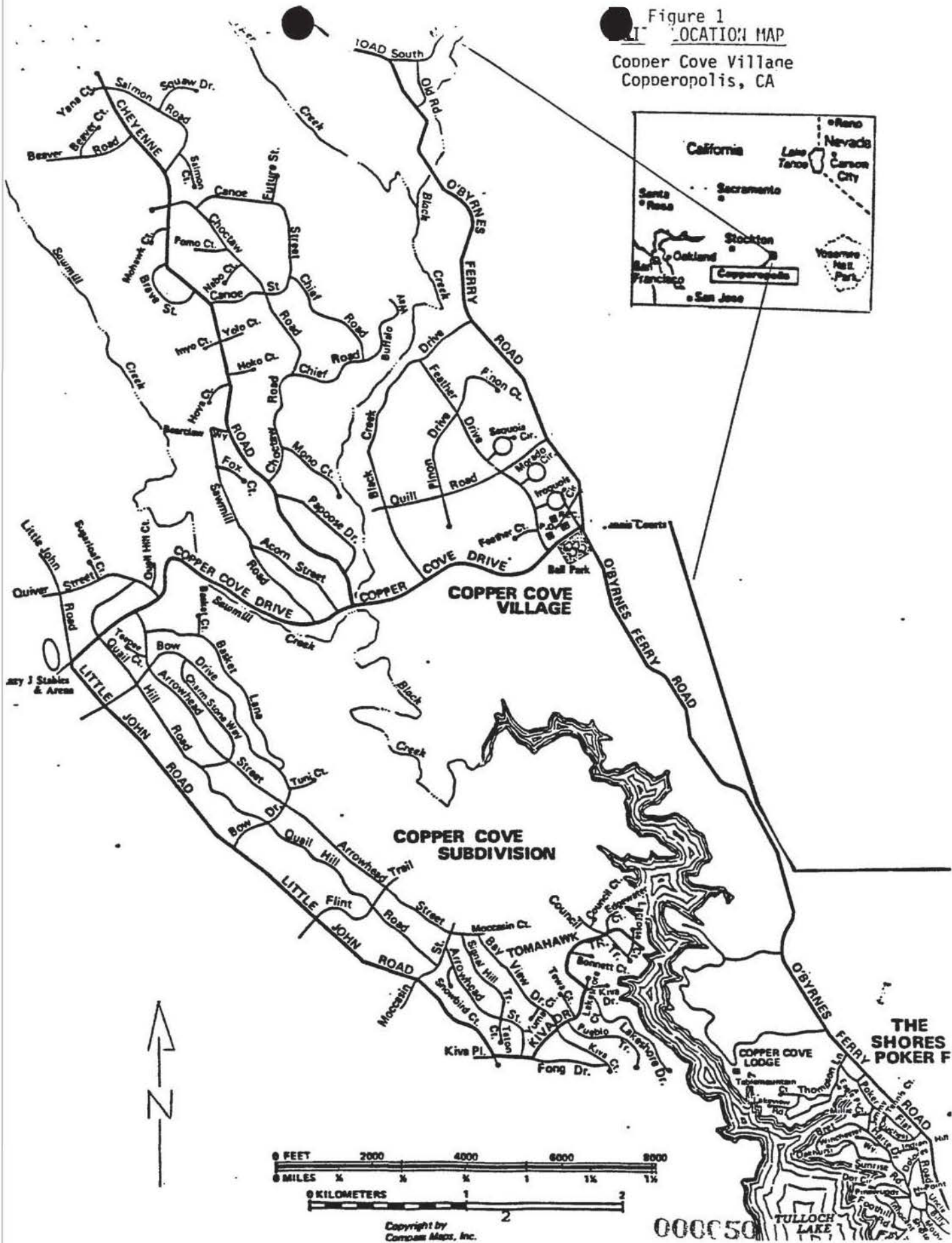
## **2.0 Sampling**

### **2.1 Preliminary Assessment Strategy**

Anticipating that the most probable areas for generating the highest amounts of airborne asbestos fibers would be those areas

000049

Figure 1  
LOCATION MAP  
Cooper Cove Villane  
Copperopolis, CA





most heavily traveled, TAT member Wolfe confined the sampling locations to the major dirt road intersections within the subdivision (Figure 2). A more thorough sampling was not possible since the assignment was limited to a one-day visit. A copy of the original PA report is included in Appendix III. Laboratory analysis of the PA samples revealed concentrations from 10 to 20 percent chrysotile asbestos, a level which exceeds the ERS action level of one percent. Unable to identify a responsible party at the onset, EPA ERS initiated a federal removal action.

## 2.2 Initiation of Federal Removal Action/Removal Action Strategy

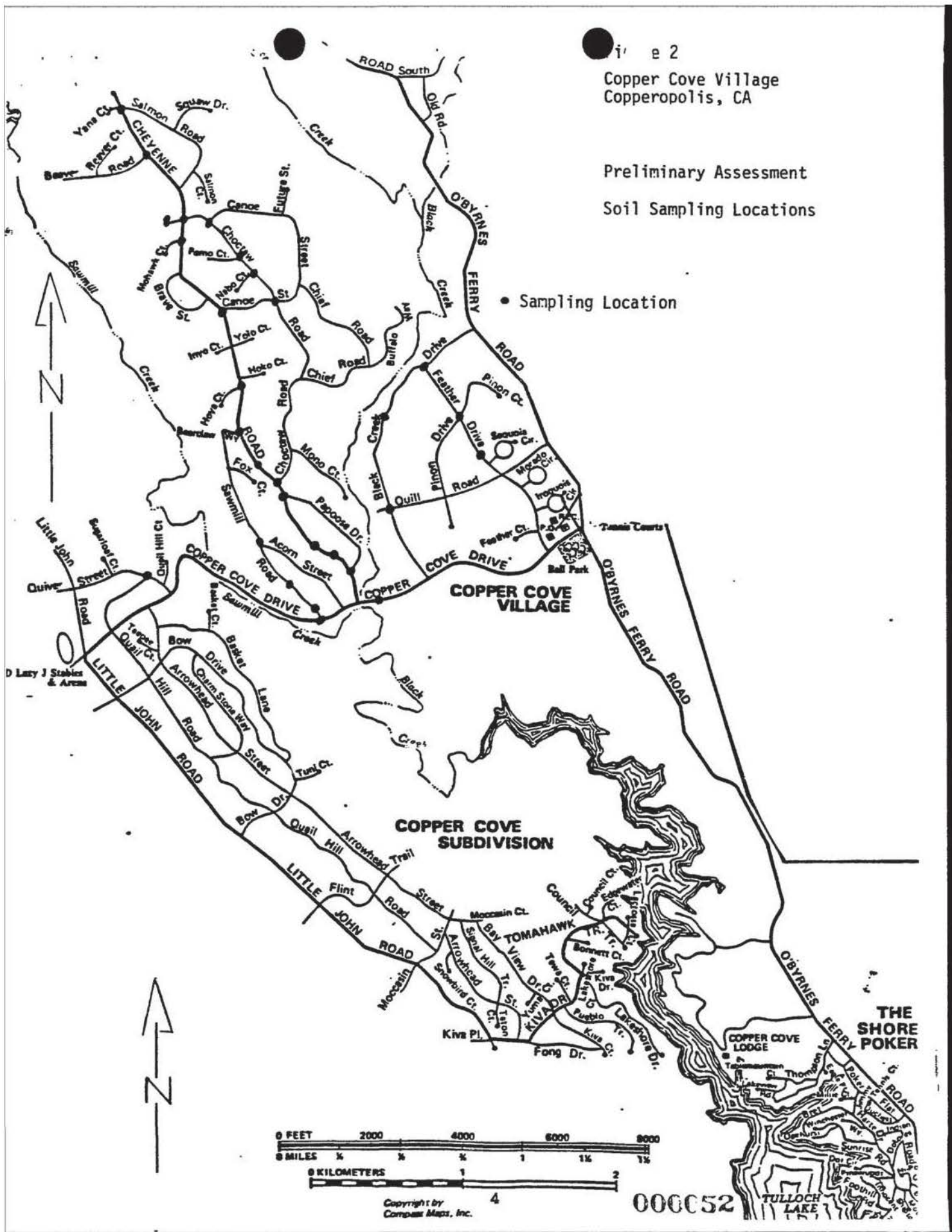
On Wednesday, April 30, 1986, EPA and TAT arrived in Copperopolis, CA to initiate immediate response action to temporarily mitigate the exposure potential of Copper Cove Village Residents to chrysotile asbestos dust. Representing the EPA was On-Scene Coordinator (OSC) Dan Shane and TAT member Gary Floyd. Personnel from the Pacific Strike Team (PST) and Reidel Environmental Services, Inc. (RES) were also on scene to assist in the removal action.

The objective of the removal action soil sampling strategy was to augment the sampling strategy implemented during the PA. Although the PA sampling provided representative data for the unpaved gravel roads, more emphasis was needed in the areas with discontinuous paved roads and selected stone driveways (Figure 3). The sampling locations chosen on the paved roads were those points where the pavement had deteriorated exposing the gravel roadbed. Driveways were sampled on account of requests by the landowners or because the driveways showed characteristics of being recently graveled (clean, loose gravel; gravel piles). In the latter case landowner permission was received prior to sampling. Also, due to the high volume of pedestrian and vehicle traffic, samples were collected on the Copperopolis Elementary School grounds (Figure 4). The most heavily traveled areas of the grounds were sampled based on the PA sampling strategy. These areas included the parking lot, playground, and school access road.

## 2.3 Sampling Technique

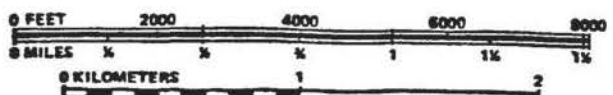
Both grab samples and composite samples were collected during the sampling process depending on the characteristics of the soil medium.

Individual grab samples were collected from areas of limited size (e.g. potholes in pavement) in which the soil compositions appeared to be homogeneous throughout the sample area. Composite samples, on the other hand, were collected from larger areas (e.g. major intersection, playground) with soils exhibiting heterogeneous characteristics. The composite samples were collected to produce a limited number of representative samples.



i' e 2  
 Copper Cove Village  
 Copperopolis, CA  
 Preliminary Assessment  
 Soil Sampling Locations

• Sampling Location



Copyright by  
 Compass Maps, Inc.

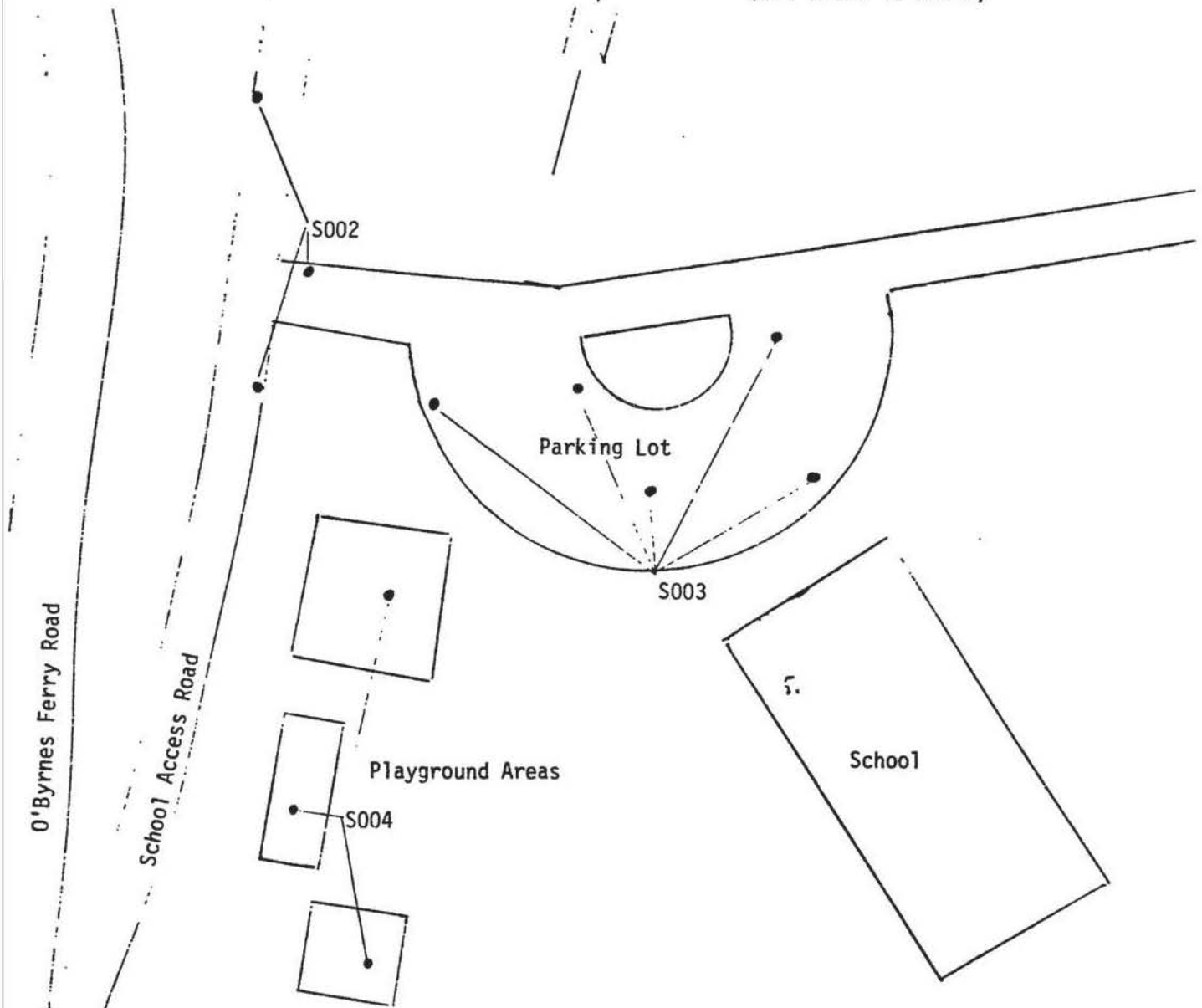
000052





Figure 4

Copperopolis Elementary School  
Soil Sampling Locations  
(not drawn to scale)





In collecting both the grab and composite bulk samples, a six inch area of the soil surface was first cleared of vegetation and debris using a clean garden trowel and the underlying soil was then removed to a three inch depth. Sample locations were photographed and sketched, and pertinent sample data was documented. In the case of collecting a grab sample, the excavated soil was placed directly into a clean, numbered sample (VOA) jar and sealed. For composite samples, the soil from the first point in the sampling area was placed into a clean cardboard bucket as were the subsequent samples taken from that same sample area. Having all the individual samples from the same area in one bucket, the material was then homogenized by mixing with the trowel and placed into a clean, numbered sample jar and sealed. The trowel and bucket were then rinsed clean using acetone to prepare the instruments for the next sampling. Collection of surface gravel samples, whether grab or composite samples, was accomplished in the same manner excluding the necessity to dig to a three inch depth. Soil sampling was conducted in Level "C" protective clothing which included an air purifying respirator to prevent dust inhalation.

#### 2.4 Field Sampling Results

All samples collected from Copper Cove Village road or driveway surfaces (13 of 17 samples; 17%) revealed laboratory analysis results of 1% to 50% chrysotile asbestos by volume.

~~The Center for Disease Control (CDC) has determined that one percent asbestos content by volume in soils is capable of generating harmful airborne asbestos concentrations.~~ This criteria, coupled with the laboratory analysis results of the PA samples (Appendix III) established the critical action level for removal action.

Laboratory analysis for all samples collected from the Copperopolis Elementary School grounds revealed asbestos concentrations of less than one percent by volume or none detected. The composite soil sample collected at the Home Owners' Association Park also revealed no chrysotile asbestos detected. Finally, one grab sample was taken from Table Mountain quarry outside of Copperopolis and revealed an asbestos concentration of 10-20 percent. This sample was collected randomly for analysis since Table Mountain quarry may be a potential source of the Copper Cove Village roadbed material.

The primary matrix materials for all soil/gravel samples collected consisted of clay particles, quartz, and feldspar. A data summary for all soil samples collected during this project is provided in Table 1.



### **3.0 Laboratory Procedures**

#### **3.1 Analytical Techniques**

##### **3.1.1 Soil Samples**

Optical polarized light microscopy, transmission electron microscopy, scanning electron microscopy, and x-ray diffraction can be employed to detect asbestos fiber concentrations in air, water or soil media. All methods are quantitative except x-ray diffraction which is a qualitative analysis with regard to bulk samples such as those collected at Copperopolis. Of these analytical techniques, transmission electron microscopy (TEM) is the most discriminating method and also the most expensive and time consuming. Accurate detection limits for TEM depend largely upon fiber morphology, optical properties, and the sampling media. Although analysis by polarized light microscopy does not provide results to the same quantitative degree (1% by volume) that TEM does, polarized light microscopy analysis can be completed in a shorter turnaround time and for a lower cost.

##### **3.1.2 Air Samples**

Air samples are typically collected by drawing a known volume of air through a sampling cassette containing a cellulose filter. Fibrous material subsequently accumulates on the filter membrane which is then analyzed by phase-contrast microscopy. Phase-contrast microscopy does not discriminate between fiber types. Asbestos fibers, wood fibers, cellulose, and fiberglass are counted as one fiber type if the fiber length to width ratio is three to one or greater. This unbiased detection capability must be considered when interpreting air sampling data obtained by phase-contrast microscopy. Air samples can also be analyzed by TEM which again would provide better quantitative data with respect to fiber type, but would result in a longer turnaround time and greater expense.

#### **3.2 Rationale for Selected Analytical Techniques**

Soil samples collected at Copperopolis were analyzed for asbestos using polarized light microscopy. This analytical technique was chosen based on cost, turnaround time, and detection limits.

All except one of the air sampling cassettes were analyzed by phase-contrast microscopy since a detection limit of .001 fiber/cc could be achieved (2000-3000 l total air volume/filter), and interfering fibrous material was not characteristic of this area. Also, a faster turnaround time can be realized at lower analytical costs when using phase-contrast microscopy. Sample TA001 was the exception to this analytical technique. This sample was analyzed for asbestos using TEM. This analytical method was chosen for this particular sample because this

Table 1  
SOIL SAMPLING DATA  
SUMMARY SHEET  
COPPEROPOLIS, CA

Sample #	Time	Date	Location	Comments	C	G	LAB ANALYSIS	
							Asbestos % Type	Other Components % Type
S001	1200	05/01/86	Quill at Black Creek Road	lt. gray, powder, fibers evident	X		30-40 chrysotile	60-70 antigorite clay particles misc. particles
S002	1132	05/01/86	school access road	lt. brown, sandy, red clay mix	X		<1 chrysotile	99+ quartz feldspar clay particles fe-oxide misc. particles
S003	1127	05/01/86	school parking lot	lt. brown, sandy	X		<1 none detected	100 quartz feldspar clay particles fe-oxide misc. particles
S004	1115	05-01-86	school playground	red sandy clay, gravel	X		<1 chrysotile	99+ quartz feldspar clay particles fe-oxide misc. particles
S005	1235	05-01-86	(b) (6)	lt. brown, dry, sandy with gravel	X		30-40 chrysotile	60-70 antigorite clay particles feldspar misc. particles
S006	1145	05/01/86	D Lazy J Stables	lt. brown, fine	X		5-10 chrysotile	90-95 quartz feldspar fe-oxide clay particles antigorite misc. particles
S007	1130	05/01/86	Basket Lane & Basket Ct. intersection	lt. gray, powder, with gravel	X		40-50 chrysotile	50-60 antigorite clay particles misc. particles

C - Composite  
G - Grab

000057



Table 1 (continued)  
SOIL SAMPLING DATA  
SUMMARY SHEET  
COPPEROPOLIS, CA

Sample #	Time	Date	Location	Comments	C	G	LAB ANALYSIS	
							Asbestos % Type	Other Components % Type
S008	1239	05/01/86	Basket Lane & Bow Drive	lt. gray, powder, w/gravel an exposed serpentine.	X		30-40 chrysotile	60-70 quartz feldspar antigorite misc. particles
S009	1145	05/01/86	Quiver Street & Little John Road	gravel w/lt. gray soil	X		30-40 chrysotile	60-70 quartz feldspar antigorite misc. particles
S010	1152	05/01/86	Basket Lane & Arrow Head	gravel w/lt. gray soil	X		40-50 chrysotile	50-60 quartz feldspar antigorite misc. particles
S011	1205	05/01/86	Quiver Street & Quail Hill Road	fine, gray powder with gravel	X		20-30 chrysotile	70-80 quartz feldspar antigorite misc. particles
S012	1726	05/01/86	Home Owners Association Park	reddish brown, clay & sand	X		<1 none detected	100 quartz feldspar clay particles misc. particles
S015	1527	05/05/86	3 pts. along Chief Road	To 3" depth, lt. gray, w gravel	X		40-50 chrysotile	50-60 antigorite clay particles feldspar misc. particles
S019	0759	05/09/86	(b) (6)	H. Dillashaw Construction hauled in stone	X		10-20 chrysotile	80-90 antigorite lizardite clay particles feldspar quartz misc. particles

C - Composite  
G - Grab

000058

Table 1 (continued)  
SOIL SAMPLING DATA  
SUMMARY SHEET  
COPPEROPOLIS, CA

Sample #	Time	Date	Location	Comments	C	G	LAB ANALYSIS	
							% Asbestos Type	% Other Components Type
S020	1630	05/09/86	(b) (6)	from driveway of (b) (6)	X		5-10 chrysotiel	90-95 antigorite lizardite clay particles feldspar quartz misc. particles iron oxides
S021	1640	05/09/86	(b) (6)	from driveway of (b) (6)	X		3-5 chrysotile	95-97 antigorite lizardite clay particles feldspar quartz misc. particles
S022	*	05/20/86	Table Mountain Quarry	Small quarry on west side	X		10-20 chrysotile	80-90 magnetite antigorite misc. particles

C - Composite

G - Grab

\* - Sample collected by EPA at unknown time

000059



cassette had purposely been subjected to a test scenario (sec. 5.3.1) which attempted to simulate worst case dust emissions. In this scenario the possibility existed that fibers with an aspect ratio of less than three to one would be generated due to the abrasive action of heavy vehicle traffic. TEM was selected based on the sensitivity and ability of this analytical technique to detect smaller fibers.

### 3.3 Quality Assurance/Quality Control (QA/QC)

Maintaining the quality of the sampling regimes, from field collection to laboratory analysis, was a primary consideration that ensured reliable data. One blind blank was submitted for analysis for every ten air samples and fractions of ten. In addition, air samples were collected from upwind control points during each sampling circuit. All samples collected at Copperopolis were logged and a strict chain of custody procedure was followed. The contracted laboratory examined each bulk soil sample submitted under low magnification to ensure that the sample was homogeneous and to make a preliminary identification of fibers present. Next, quantification of those fibers was obtained through higher magnification (400-450x) and manual counting. Every tenth soil sample was re-analyzed in the laboratory as an internal QA/QC procedure. This same procedure was applied to air samples as well. Re-analyzed results were required show a 95% or greater confidence level to be acceptable.

### 4.0 Disposal Alternative for Asbestos

There are three acceptable methods for the disposal of asbestos according to EPA. Those methods include: 1) excavation and transportation to an off-site landfill, 2) burial in an on-site landfill, or 3) in-situ capping. Several factors influenced which method would be most applicable to the Copperopolis site. As mentioned earlier, the source of the asbestos-laden dust was approximately 16 miles of unimproved roads constructed using serpentine rock. Removal of such an excessive volume of roadbed material (estimated 850,000 cu. yd.) would not only be extremely expensive and impractical, but the process would increase the risk of emitting additional asbestos fibers into the air and prove to be very disruptive to the residents of Copper Cove Village. The characteristic low relative humidity of this area and subsequent reduced ability of the air to trap fibers would only compound the dust problem. Considering these factors, the most viable alternative was to cap the road surfaces.



## 5.0 Immediate Removal Action

### 5.1 Decision to Apply Chip Seal

The initial plan of action was to apply a lignosulfate coating to the more than 16 miles of road surface to act as a dust suppressant and bonding agent. However, after making a comparison of lignosulfate and chip seal based on initial cost, ten-year total cost, annual efficiency of percent chrysotile controlled, frequency of application, ease of driving, and adverse environmental impacts (Table 2), OSC Shane made the decision to use chip seal.

### 5.2 Chip Seal Application

#### 5.2.1. Phase I (April 30 - May 12, 1986)

All roads in Copper Cove Village intended for chip seal application had to first undergo a moderate preparation process. This preparation involved grading to a depth of 3 to 4 inches which eliminated potholes and uneven surfaces and provided a center crown; clearing existing ditches since chip seal was to extend the full width of the road and to better accommodate runoff water, and finally to roll all graded surfaces to compact the loose aggregate and insure an even surface. A water tank truck preceeded the grading operation and continuously saturated the road surface to reduce dust emissions (the effectiveness of water application will be discussed in 5.3). After the roadbed preparation was completed, a slow curing oil (SC-70) was applied as the initial dust suppressant and bonding agent. At the owner's request, private driveways received this same treatment. The last step of Phase I involved the application of a rolling rock dust to oiled road surfaces to facilitate drying and to minimize splashing as a result of vehicle traffic.

Contractors involved with this phase of the road preparation and oil application included: Tri-County Construction (initial grading, rolling, watering), Reed and Graham (supplied and spread oil), Triple C (supplied rock dust), Glen Wilson Trucking (hailed rock dust to site), and Henley (applied rock dust).

After the initial roadbed preparation and oil application phase was completed, a ten day curing period followed before the second and final phase of the removal action commenced.

#### 5.2.2 Phase II (May 20 - 22, 1986)

During this phase of the capping process a single layer of rock chips was applied to 8.6 miles of secondary roadways which is projected to remain effective for up to 3 years. A double layer was applied to 8.1 miles of primary roadways including the first one half mile of Cheyenne, the main thoroughfare of the subdivision, which increased the road surface longevity and effectiveness for up to 10 years (Figure 5).

TABLE 2 A COMPARISON OF CONTROL ALTERNATIVES  
FOR REDUCING CHRYSOTILE EMISSIONS FROM UNPAVED ROADS<sup>1-18</sup>

Control option	Initial cost \$1,000/mi <sup>a</sup>	Annual maintenance cost \$1,000/mi	Ten-year total cost \$1,000/mi	Ten-year total cost \$1,000/mi (midrange)	Annual efficiency percent chrysotile controlled <sup>b</sup>	Frequency of application <sup>c</sup>	Adverse environmental impacts	Ease of driving
1. Stone replacement	32	0.6-2.5	37-55	46	100	1 time	None	No change
2. Single-coat chip seal	5-8	0.8	21-30	26	90-99.9	3 times/10 yr	None	Excellent
3. Triple-coat chip seal	20-37	0.2	22-39	31	90-99.9	1 time/10 yr	None	Excellent
4. Petroleum products	4-11	4-11	40-110	75	60-85	2 times/yr	None	Good
5. Lignosulfonate <sup>d</sup>	3.6-8.5	3.6-8.5	36-85	61	60-80	1 time/yr	None	Good
6. Calcium chloride	3-5	3-5	30-50	40	60-80	3 times/yr	Yes	Good
7. Water and wetting agents	27-42	27-42	270-420	345	40-60	3 times/wk	None	Fair
8. Speed control 30-10 mph	0	0	96-304 <sup>e</sup>	199	66	--	None	No change
9. Speed control 30-20 mph	0	0	24-76 <sup>e</sup>	50	33	--	None	No change
10. Stone replenishment	21	0.6-2.5	26-44	35	f	1 time/yr	None	No change

<sup>a</sup>Costs include labor and surface preparation. All cost figures are in 1979 dollars.

<sup>b</sup>Assumed to be proportional to efficiency in controlling dust emissions.

<sup>c</sup>Applications necessary to maintain annual efficiency rating.

<sup>d</sup>The cost of lignosulfonates may be significantly reduced if a local supply can be secured at nominal cost.

<sup>e</sup>Estimated social cost of time "lost" by traveling slower. An average daily traffic volume of 100 vehicles was assumed. The range represents the value of lost time for individual (\$4 per car-hour) and commercial drivers (\$12.50 per vehicle-hour).

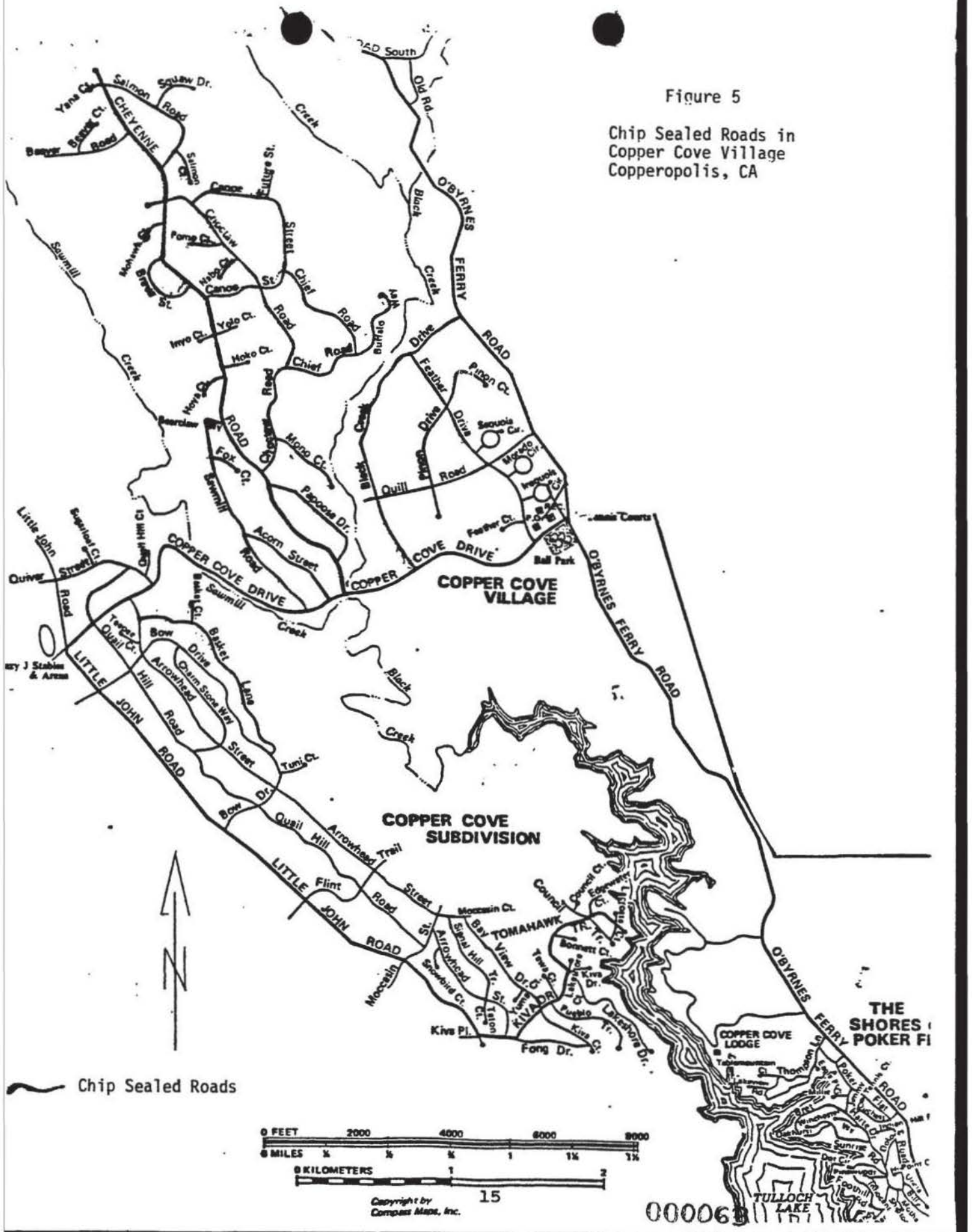
<sup>f</sup>Not known.

Assessment and Control of Chrysotile Asbestos Emissions from Unpaved Roads, EPA Document 450/3-81-006



Figure 5

Chip Sealed Roads in  
Copper Cove Village  
Copperopolis, CA



The ~~chipping process~~ first involved the application of an asphalt anionic emulsion followed by another application consisting of a hot asphalt cationic emulsion and crushed lava rock. This method insured better bonding results, a higher quality seal, and consequently provided maximum asbestos dust suppression. Also during this phase, culverts were emplaced at the entrance to a few private driveways at the owners' request.

Contractors involved with this phase of the chip sealing operation included: Bob Harkrader Trucking (hauled lava chips to site) Don Milner (applied lava chips and rolled road), Ray Fuller (supplied culverts), and A&L Ready Mix (supplied concrete for culverts).

### 5.3 Air Monitoring

Air monitoring for asbestos fibers was conducted concurrently with all road preparation, and oil application work (Phase I). EPA Document No. 560-15-85-024 established exposure guidelines which state that ambient air concentrations of asbestos fibers which exceed .01 fibers/cc could result in adverse health effects. Relative to these guidelines, air monitoring was conducted with the objective of documenting not only generated levels of asbestos dust exceeding the .01 fibers/cc limit, but all generated levels. The purpose of continuously wetting the road surfaces during the removal operation was to reduce or eliminate airborne asbestos fiber generation. Table 3 summarizes all air monitoring data for this project. From the table one can see that asbestos fiber generation was considerably less than .01 fibers/cc. Variable or downwind asbestos concentrations ranged from was 0.00033 - 0.0015 fibers/cc, while the range for the control or upwind samples was 0.00033 - <0.00036.

#### 5.3.1 Test Scenario

In conjunction with the continuous air monitoring of the road work, a test scenario was conducted in order to obtain results that would simulate the generation of airborne chrysotile asbestos fibers due to normal to heavy vehicle traffic in Copper Cove Village. A similar test was first conducted by EPA in 1979 (EPA-450/3-81-006).

A 0.3 mile section of Chief Road (Figure 6) was chosen for the test due to the representative road composition. The fact that this section of road had been dry for quite some time and that wind direction was typically perpendicular to the road made the area ideal. Five air sampling locations were chosen at equidistant intervals along the entire test section, two of which were variable (downwind) locations and two were control (upwind) locations (pump #5 blew a fuse early on in the test). A weather station was also used to monitor wind direction, wind speed, and temperature at 30 minute intervals throughout the entire two hour test.



Table 3  
AIR SAMPLING DATA  
SUMMARY SHEET  
COPPERPOLIS, CA

Sample #	Start Time	Date	Setting (l/min)	Air Pump Location	Comments	C	V	Stop Time	LAB ANALYSIS		
									ASBESTOS FIBER COUNT Fibers/Filter	Fibers/cc	VOLUME liter
A001	1124	05/05/86	10 l/min	On Papoose Dr. at corner above S. int. of Papoose & Cheyenne	SE side of road; downwind	X		1632	<1100.*	<0.00036	3000.
A002	1129	05/05/86	10 l/min	same as A001	NW side of road; upwind	X		1637	<1100.*	<0.00036	3000.
A003	1144	05/05/86	10 l/min	On east side of Cheyenne at int. of Bearclaw Way	downwind		X	1645	<1100.*	<0.00036	3000.
A004	1200	05/05/86	10 l/min	On east side of Cheyenne at int. of Canoe	downwind		X	1700	<1100.*	<0.00036	3000.
A005	1215	05/05/86	10 l/min	On east side of Cheyenne at int. of Salmon. (S. entrance)	downwind		X	1816	<1100.*	<0.00052	2070.
A006	0737	05/06/86	10 l/min	On Papoose Dr. at corner above S. int. of Papoose & Cheyenne	SE side of road; upwind	X		1246	<1100.*	<0.00036	3000.
A007	0737	05/06/86	10 l/min	same as A006	NW side of road upwind	X		1246	<1100.*	<0.00036	3000.
A008	0802	05/06/86	10 l/min	Canoe at Choctaw (S)	downwind		X	1254	<1100.*	<0.00036	3000.
A009	0815	05/06/86	10 l/min	Center Pt. of Canoe	downwind		X	1256	<1100.*	<0.00036	3000.

C - Control  
V - Variable

\* - Phase Contrast

Note: Threshold value (TLV) chrysotile asbestos (CAS 12001-29-5) is 2.0 fibers/cc.  
(fibers longer than 5 micros with an aspect ration  $\geq$  3:1) ACGIH 1985

000065

Table 3 (continued)  
AIR SAMPLING DATA  
SUMMARY SHEET  
COPPEROPOLIS, CA

Sample #	Start Time	Date	Setting (l/min)	Air Pump Location	Comments	C	V	Stop Time	LAB ANALYSIS		
									ASBESTOS FIBER COUNT Fibers/Filter	Fibers/cc	VOLUME liter
A010	0828	05/06/86	10 l/min	Canoe at Choctaw (N)	downwind; 10 l/min.		X	1258	<1100.*	<0.00034	3000.
A011	0829	05/07/86	10 l/min	Northern most end of Cheyenne	upwind; control station for ambient readings		X	1332	<1100.*	<0.00034	3000.
A012	0819	05/07/86	10 l/min	center pt. of Brave St.	downwind		X	1325	<1100.*	<0.00034	3000.
A013	0801	05/07/86	10 l/min	int. of Cheif Rd. & Canoe St.	down at 0910 start up at 1208; downwind		X	1500	<1100.*	<0.00034	3000.
A014	0907	05/07/86	10 l/min	on Cheyenne between Inyo Ct. & Hoya St.	downwind		X	1358	<1100.*	<0.00034	3000.
A015	0901	05/07/86	10 l/min	Choctaw at Cheyenne	downwind		X	1358	<1100.*	<0.00034	3000.
A016	0954	05/08/86	10 l/min	Entrance to Home Owners Assoc. Park on Black Creek Rd.	NW side of road downwind; high traffic volume		X	1535	4500.*	0.0015	3000.
A017	1120	05/08/86	10 l/min	Mid point between Quill & Feather on Black Creek Rd.	down at 1320. Start at 1445 off at 1755; downwind		X	1750	<1000.*	<0.00034	3000.
A018	1128	05/08/86	10 l/min	Quill Rd. at Black Creek Rd.	off at 1200 on at 1210; downwind		X	1758	2000.*	0.00067	3000.

C - Control  
V - Variable

000066

Table 3 (continued)  
AIR SAMPLING DATA  
SUMMARY SHEET  
COPPERFOLIS, CA

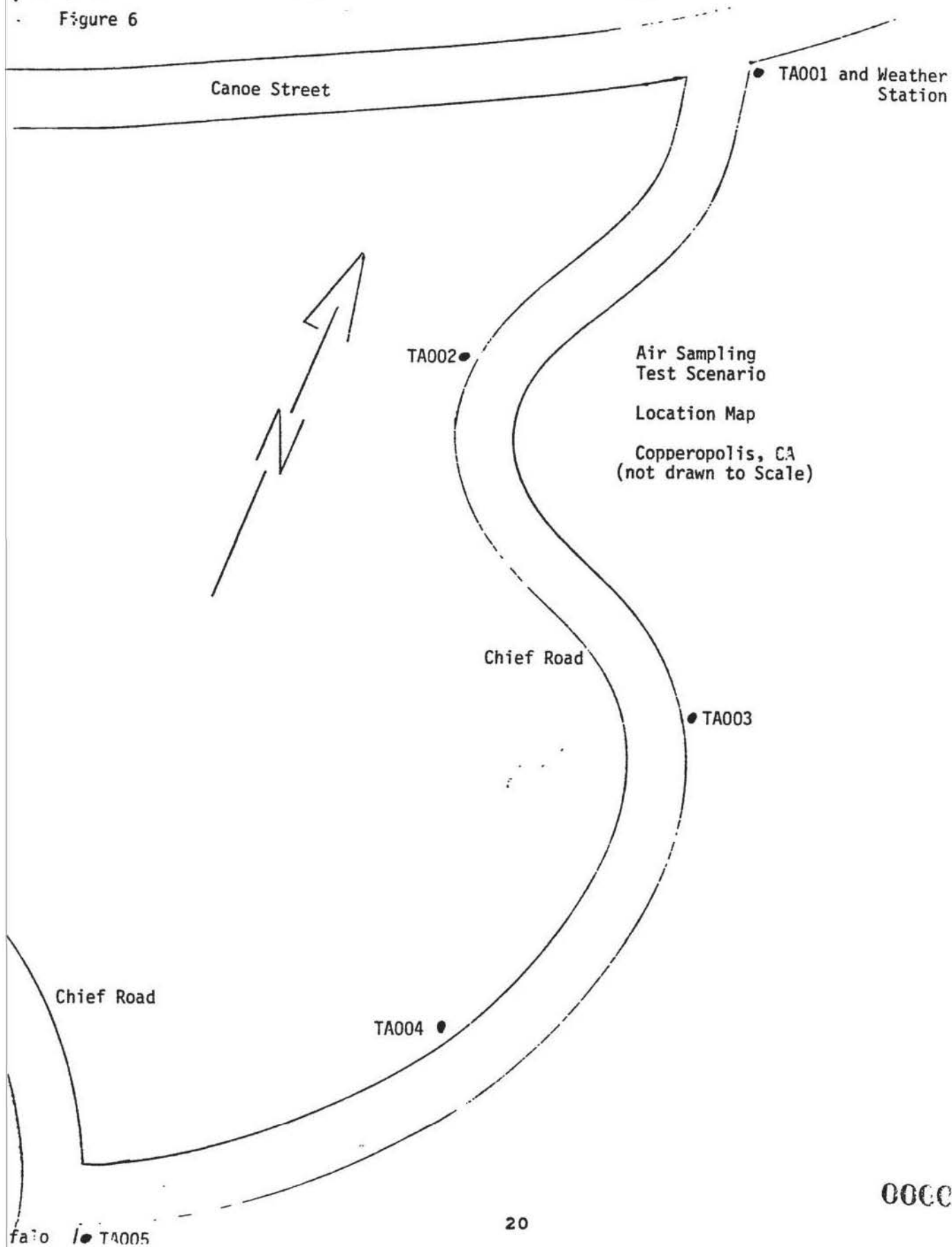
Sample #	Start Time	Date	Setting (l/min)	Air Pump Location	Comments	C	V	Stop Time	LAB ANALYSIS		
									ASBESTOS FIBER COUNT Fibers/Filter	Fibers/cc	VOLUME liter
A019	0706	05/09/86	10 l/min	on Feather approx. 100' N. of Sequoia Circle	upwind	X		1220	1000.*	0.00033	3000.
A020	0713	05/09/86	10 l/min	SW side of int. of Feather & Pinion	downwind		X	1230	1000.*	0.00033	3000.
A021	0722	05/09/86	10 l/min	inside of hairpin turn on Pinion.	downwind		X	1235	1000.*	0.00033	3000.
A022	N/A	05/09/86	N/A	N/A - blank	control blank	X		1300	1000.*	0.00033	0.0

C - Control  
V - Variable

000067



Figure 6



000068

Air sampling pumps were calibrated in the field to draw 16.7 liters of air per minute providing a total sample volume of 2004 liters for each pump. This permitted accurate laboratory analysis by phase-contrast microscopy.

The test involved driving a vehicle at a representative speed (24 mph) and making 80 consecutive passes of the sampling stations. All vehicle windows and vents remained closed to avoid inhalation of any generated fibers by the driver. At the end of the test, all samples were collected and logged under chain of custody.

Samples TA002 through TA005 (blank) were analyzed by phase-contrast microscopy as mentioned earlier, while sample TA001 was analyzed by TEM to serve as a comparison to the phase-contrast results (Table 4). All test sample analyses showed elevated asbestos fiber concentrations when compared to those collected concurrent with road work. Two of the test samples exceeded the .01 fibers/cc EPA exposure guidelines which suggest that driving on unpaved roads in Copper Cove Village could have posed a health threat, and that watering roads prior to preparation work significantly aided in reducing airborne asbestos fibers.

#### 5.4 Weather Monitoring

Monitoring of wind direction, wind speed and temperature was conducted concurrently with the road preparation and oil application phase of the project. Monitoring of the prevailing wind direction assisted in the selection of air sampling station locations. Weather station data summary is provided in Table 5. A graphic representation can be found in Table 6.

#### 5.5 Cost Monitoring

Daily cost monitoring was completed on site by PST member G. Perry with assistance from TAT member G. Floyd using the EPA's cost monitoring software and a personal computer. Total daily costs were reviewed and approved by OSC D. Shane, the PST Commanding Officer, and RES's project manager at the close of work each day.

#### 6.0 Remedial Action

With removal action completed by the EPA Emergency Response Section, the project has been turned over to the EPA Enforcement Response Section for remedial action. This initiative will involve further identification of potential responsible parties and possible removal cost recovery.

000069



Table 4  
Test Scenario  
AIR SAMPLING DATA  
SUMMARY SHEET  
COPPERFOLIS, CA

Sample #	Start Time	Date	Setting (l/min)	Air Pump Location	Comments	C	V	Stop Time	LAB ANALYSIS		VOLUME liter
									ASBESTOS FIBER COUNT Fibers/Filter	Fibers/cc	
TA001	1600	05/07/86	16.7l/min	Canoe at Chief	downwind; 80 passes w/vehicle avg. 12 mph 2 hrs. duration	X		1807	** 120,000ng/cm	0.90** 0.06***	2004.
TA002	1610	05/07/86	16.7l/min	3/10 mi. NW of TA005	upwind	X		1807	18000.*	0.0090	2004.
TA003	1614	05/07/86	16.7l/min	2/10 mi. NW of TA005	downwind	X		1827	2000.*	0.0010	2004.
TA004	1616	05/07/86	16.7l/min	1/10 min NW of TA005	upwind	X		1828	35000.*	0.17	2004.
TA005	N/A	05/07/86	N/A	N/A - blank	blank	X		N/A	<1000.*	<0.00050	0.0
TA006	N/A	05/15/86	N/A	N/A - blank	blank	X		N/A	Not Analyzed	Not Analyzed	0.0

Note: Threshold limit value (TLV) chrysotile asbestos (CAS 12001-29-5) is 2.0 fibers/cc.  
(fibers longer than 5 microns with an aspect ratio  $\geq$  3:1) ACGIH 1985

\* Phase-Contrast analysis

\*\* TEM analysis

\*\*\* Phase-Contrast equivalent

C - Control

V - Variable

000070

Table 5  
Weather Station Data  
Summary Sheet  
Copperopolis, CA

TIME	DATE	LOCATION	WEATHER STATION INFORMATION		
			Wind Speed	Temperature	Wind Direction
1038	05/03/86	MCP - Copper Cove Dr. at Cheyenne	<1 mph	60°F w/rain	Northeast
1246	05/03/86	MCP - Copper Cove Dr. at Cheyenne	2 mph	61°F overcast	East - Southeast
1414	05/03/86	MCP - Copper Cove Dr. at Cheyenne	1-2 mph	56°F w/rain	North - Northeast
1501	05/03/86	MCP - Copper Cove Dr. at Cheyenne	8 mph	55°F w/rain	North - Northeast
0758	05/05/86	MCP - Copper Cove Dr. at Cheyenne	<1 mph	53°F overcast	North - Northeast
1047	05/05/86	MCP - Copper Cove Dr. at Cheyenne	1-2 mph	59°F overcast	North
1242	05/05/86	MCP - Copper Cove Dr. at Cheyenne	<1 mph	67°F overcast	North - Northeast
1411	05/05/86	MCP - Copper Cove Dr. at Cheyenne	1-2 mph 2-6 mph	72°F overcast	North
1500	05/05/86	MCP - Copper Cove Dr. at Cheyenne	2 mph	69°F	North
1610	05/05/86	MCP - Copper Cove Dr. at Cheyenne	4-6 mph	66°F	North
1620	05/05/86	MCP - Copper Cove Dr. at Cheyenne	0-2 mph	65°F	Southwest
1735	05/05/86	MCP - Copper Cove Dr. at Cheyenne	1-2 mph	63°F	North

000C71



Table 5 (continued)  
Weather Station Data  
Summary Sheet  
Copperopolis, CA

TIME	DATE	LOCATION	WEATHER STATION INFORMATION		
			Wind Speed	Temperature	Wind Direction
1849	05/05/86	MCP - Copper Cove Dr. at Cheyenne	1-2 mph	61°F	North
0852	05/06/86	MCP - Copper Cove Dr. at Cheyenne	1-2 mph	49°F	North
0949	05/06/86	MCP - Copper Cove Dr. at Cheyenne	1-2 mph	51°F	East
1129	05/06/86	MCP - Copper Cove Dr. at Cheyenne	4-6 mph	58°F	North
1241	05/06/86	MCP - Copper Cove Dr. at Cheyenne	14 pmh	63°F	North
1750	05/06/86	MCP - Copper Cove Dr. at Cheyenne	1-2 mph	60°F	North - Northeast
1919	05/06/86	MCP - Copper Cove Dr. at Cheyenne	1-2 mph	55°F	North - Northwest
0729	05/07/86	MCP - Copper Cove Dr. at Cheyenne	<1 mph	58°F	North - Northeast
0845	05/07/86	MCP - Copper Cove Dr. at Cheyenne	<1 mph	70°F	North - Northeast
0933	05/07/86	MCP - Copper Cove Dr. at Cheyenne	4 mph	74°F	North - Northeast Clear
1046	05/07/86	MCP - Copper Cove Dr. at Cheyenne	<1 mph	76°F	North - Northeast Clear
1235	05/07/86	MCP - Copper Cove Dr. at Cheyenne	1-2 mph	80°F	North Clear

000072

Table 5 (continued)  
Weather Station Data  
Summary Sheet  
Copperopolis, CA

TIME	DATE	LOCATION	WEATHER STATION INFORMATION		
			Wind Speed	Temperature	Wind Direction
1605	05/07/86	Chief and Chactaw (test area)	2-4 mph	86°F	Northeast Clear
1630	05/07/86	Chief and Chactaw (test area)	6 mph	82°F	Northeast Clear
1704	05/07/86	Chief and Chactaw (test area)	10 mph	80°F	East - Northeast Clear
1733	05/07/86	Chief and Chactaw (test area)	2 mph	84°F	Northeast Clear
1809	05/07/86	Chief and Chactaw (test area)	4 mph	78°F	North - Northeast Clear
0752	05/08/86	MCP - at Home Owners Association Park	<1 mph	61°F	North - Northeast Clear
1135	05/08/86	MCP - at Home Owners Association Park	2 mph	76°F	West - Northwest Clear
1201	05/08/86	MCP - at Home Owners Association Park	4 mph	76°F	Northwest Clear
1406	05/08/86	MCP - at Home Owners Association Park	8 mph	90°F	North Clear
1521	05/08/86	MCP - at Home Owners Association Park	2-4 mph	94°F	North Clear
1551	05/08/86	MCP - at Home Owners Association Park	6-8 mph	95°F	North Clear
1732	05/08/86	MCP - at Home Owners Association Park	4-6 mph	87°F	North Clear

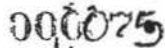
000073



Table 5 (continued)  
Weather Station Data  
Summary Sheet  
Copperopolis, CA

TIME	DATE	LOCATION	WEATHER STATION INFORMATION		
			Wind Speed	Temperature	Wind Direction
1808	05/08/86	MCP - at Home Owners Association Park	10 mph	88°F	North Clear
0654	05/09/86	MCP - at Home Owners Association Park	<1 mph	50°F	North Clear
0757	05/09/86	MCP - at Home Owners Association Park	<1 mph	70°F	North Clear
0839	05/09/86	MCP - at Home Owners Association Park	1-2 mph	76°F	North Clear
0925	05/09/86	MCP - at Home Owners Association Park	<1 mph	80°F	North - Northeast Clear
1046	05/09/86	MCP - at Home Owners Association Park	4-6 mph	85°F	South Clear
1142	05/09/86	MCP - at Home Owners Association Park	2-4 mph	85°F	South Clear
1246	05/09/86	MCP - at Home Owners Association Park	1-2 mph	89°F	East - Northeast
1401	05/09/86	MCP - at Home Owners Association Park	4-6 mph	89°F	South - Southwest
1711	05/09/86	MCP - at Home Owners Association Park	1-2 mph	95°F	West - Southwest

REVALUATING MIND DISSECTION





## 7.0 Summary

After being notified by the Copperopolis Community Board of a potential asbestos problem existing in Copper Cove Village, the EPA ERS dispatched TAT to conduct a preliminary assessment (PA) of the area. Soil samples collected during the PA were gathered from areas suspected to be the sources of airborne asbestos fiber generation such as major intersections. Laboratory analysis of the PA samples revealed concentrations from 10 to 20 percent chrysotile asbestos, which exceeds the ERS action level of one percent.

Unable to identify a responsible party at the onset, EPA ERS initiated a federal removal action which included additional soil sampling to substantiate the findings of the PA. Analysis of this second set of samples revealed asbestos concentrations as high as 50 percent. The next phase of the removal action involved road preparation and the application of a single coat of lava rock chip seal to 8.6 miles of secondary roads, and a double coat to 8.1 miles of primary roads within Copper Cove Village. Lignosulfate was originally intended to be used as the capping material, but comparison of this material to chip seal based on product longevity, initial cost, maintenance cost, and ease of driving indicated that chip seal would be the best product for the job.

Air sampling conducted concurrent with road preparation work indicated that generation of airborne asbestos fibers did not exceed the .01 fibers/cc guideline established by EPA. An air sampling test scenario was also conducted in which a representative section of unpaved road was subjected to two hours of continuous vehicle traffic. Air monitoring analysis of the test indicated that asbestos dust emissions can in fact exceed the .01 fibers/cc level as a result of driving on unpaved roads in Copper Cove Village. Daily wind speed direction, and temperature were monitored to assist in selecting air sampling station locations.

Daily costs incurred were accounted for on site at the close of each day using EPA's cost monitoring software and a personal computer. EPA ERS and TAT work was completed with the application of the chip seal. The continuing remedial work has been turned over to EPA's Enforcement Response Section which will involve further identification of potential responsible parties and possible removal cost recovery.

### Bibliography

U.S. Environmental Protection Agency. Assessment and Control of Chrysotile Asbestos Emissions from Unpaved Roads. Triangle Park, NC: EPA document 450/3-81-006, 1981.

U.S. Environmental Protection Agency. Emergency Response to Asbestos Contaminated in Alviso, CA. San Francisco, CA: EPA document TDD #098510-06; PCS #5006, 1986.

000077

APPENDIX I  
Photographic Documentation

000078



WESTON

Copper Cove Village Asbestos Site, Copperopolis, CA

PCS #5137

TDD #098604-20

Photographer: Gary Floyd

Date: 05/01/86



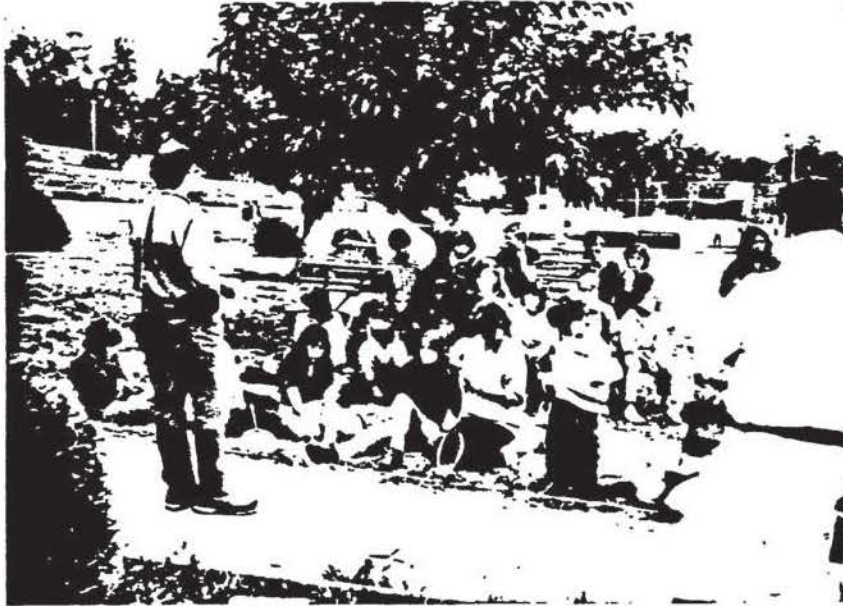
Soil sampling of the roads in Copper Cove Village.  
All sampling was conducted in level 'C' protective clothing.



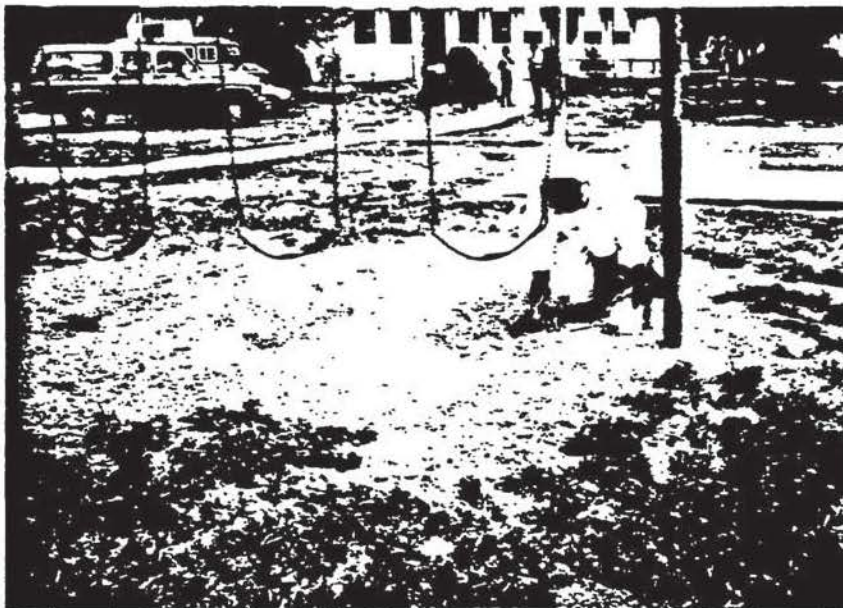
000079

**WESTON**

Copper Cove Village Asbestos Site, Copperopolis, CA  
PCS #5137 TDD #098604-20  
Photographer: Gary Floyd Date: 05/01/86



EPA OSC Dan Shane informed students at Copperopolis Elementary School of the health effects associated with asbestos inhalation and what the EPA was doing about the situation in Copperopolis.



Soil sampling on the Copperopolis Elementary School grounds.

000C80



WESTON

Copper Cove Village Asbestos Site, Copperopolis, CA  
PCS #5137 TDD #098604-20  
Photographer: Gary Floyd Date: 05/01/86



Road preparation first involved grading and leveling the surface.



The freshly graded surface was then compacted using rollers.

000C81





Copper Cove Village Asbestos Site, Copperopolis, CA  
PCS #5137 TDD #098604-20  
Photographer: Gary Floyd Date: 05/01/86



A site map board was used at the Mobile Command Post (MCP)  
to track work progress.

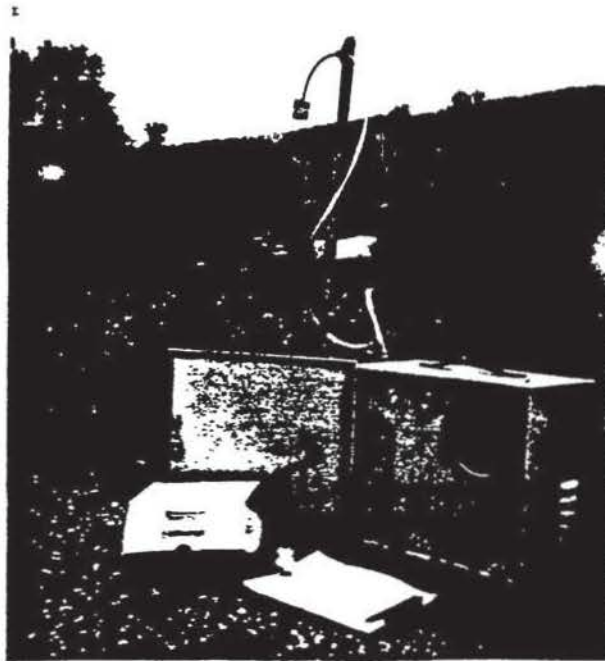
000082



Copper Cove Village Asbestos Site, Copperopolis, CA  
PCS #5137 TDD #098604-20  
Photographer: Gary Floyd Date: 05/01/86



Representative air sampling station.  
Sampling was conducted concurrent to road preparation.



Upwind (control) air sampling station.

000083

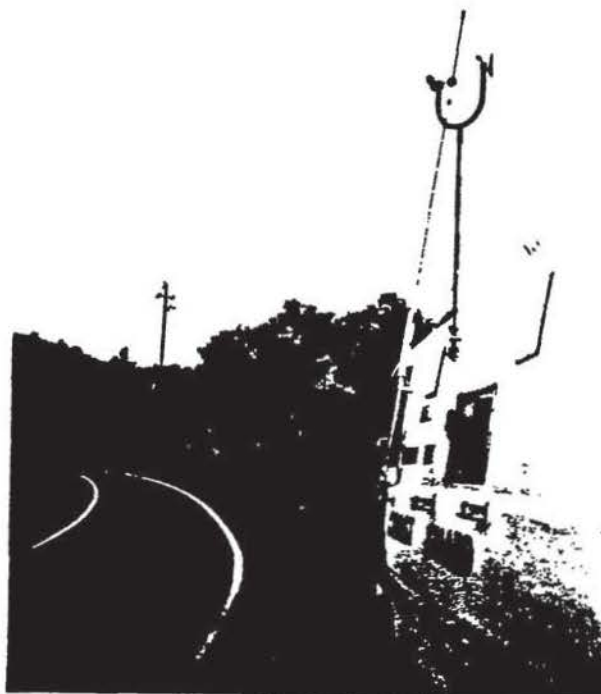


Copper Cove Village Asbestos Site, Copperopolis, CA  
PCS #5137 TDD #098604-20  
Photographer: Gary Floyd Date: 05/01/86



Strict chain of  
custody logging  
was followed for  
all samples.

A weather  
station was  
used to measure  
wind speed,  
wind direction,  
and temperature.



000084



WESTON

Copper Cove Village Asbestos Site, Copperopolis, CA

PCS #5137

TDD #098604-20

Photographer: Gary Floyd

Date: 05/01/86



Oil being transferred to spray truck for road application.



Recently oiled road prior to the application  
of blotting material.

000085

WESTON

Copper Cove Village Asbestos Site, Copperopolis, CA  
PCS #5137 TDD #098604-20  
Photographer: Gary Floyd Date: 05/01/86



Oil being transferred to spray truck for road application.



Recently oiled road prior to the application  
of blotting material.

000086

**WESTON**

Copper Cove Village Asbestos Site, Copperopolis, CA

PCS #5137

TDD #098604-20

Photographer: Gary Floyd

Date: 05/01/86



Both personnel and vehicles were decontaminated at the close of work each day.



000087



**APPENDIX II**  
**Analytical Techniques For Asbestos**

000088

## 2.3 ASBESTOS MEASUREMENT TECHNIQUES

### 2.3.1 Air Sample Analyses

Airborne particles are initially collected on the surface of a small-pore size filter through which a known volume of air is drawn. Asbestos fiber concentrations are then estimated by either phase contrast microscopy (a special application of the optical microscope) or by electron microscopy (EM). In general, asbestos fiber concentration data obtained by one of these two methods cannot be converted to a concentration determined by the other method. In phase contrast microscopy, a section of a membrane filter is viewed at 400 magnification, and all particles which have at least a 3:1 length-to-width ratio and a length of 5  $\mu\text{m}$  or greater are counted as asbestos fibers. Fibers smaller than 0.1  $\mu\text{m}$  in diameter are not visible by phase contrast microscopy. Consequently, identification of smaller size fibers which may be of biological significance is precluded. Fiber counting by phase contrast microscopy is based entirely on particle shape and is not specific for asbestos. This method is the standard method for measuring asbestos in the workplace. In cases where all fibers are smaller than 5  $\mu\text{m}$  in length or thinner than 0.1  $\mu\text{m}$  in diameter, no detectable fiber count will result.

By comparison, electron microscopy permits positive identification of asbestos fibers that are not observable by phase contrast microscopy. Chrysotile fibers are relatively easy to distinguish from other types of fibers because of their unique tubular structure. Selected area electron diffraction (SAED) and energy dispersive X-ray diffraction (EDX) are often used to substantiate fiber identification. In EM analysis, only a very small fraction of the filter is viewed at a magnification of 15,000 to 20,000 X.

When determining asbestos concentrations in airborne particulate samples by either phase contrast microscopy or EM, visible fiber counts are used to estimate the total fiber count for the whole filter. The accuracy of the calculated fiber concentration is primarily dependent upon the representativeness of the fiber population actually counted.

The method of sample preparation and fiber counting strongly influence the results obtained by EM. EPA developed a provisional methodology in

1977 that optimized various techniques for analyzing and counting airborne asbestos fibers by EM. Testing of the provisional methodology showed inter-laboratory variation in fiber concentration results to be about 20 percent for samples collected in an industrial setting and samples prepared in the laboratory.<sup>5 6</sup>

### 2.3.2 Bulk (Rock) Sample Analysis

Petrographic microscopy is the principal method for examining bulk samples (such as a rock sample). This technique is relatively straightforward and reliable for qualitative identification and characterization of crystalline substances, including asbestos.

Quantitative analysis of asbestos in a bulk sample is determined by EM. A small representative portion of rock powder is ground from a bulk sample and is uniformly dispersed onto a filter media. A small fraction of the filter is then viewed at high magnification, and asbestos mass and fiber concentrations are estimated. Careful consideration must be given to sample preparation, especially during the grinding phase. Chrysotile is usually present in microveins that tend to disintegrate into small chunks. These chunks must be given sufficient grinding time to divide into free fibers. A methodology for analysis of asbestos in rock samples was published by EPA in December 1978.<sup>7</sup> The methodology is summarized in Appendix B.

000090



FORMULA: various

# FIBERS

M.W.: various

METHOD 7400  
ISSUED: 2/15/84

OSHA: 0.5 asbestos fibers (f/m) long/m

PROPERTIES: soft  
fibrous

NIOSH: 0.1 asbestos f/mL [1]; 3 glass fibers ( $>10 \mu\text{m} \times <3.5 \mu\text{m}$ )/mL [2]

ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, f/mL

SYNONYMS: asbestos (actinolite [CAS #13768-00-8], grunerite (amosite) [CAS #12172-73-5], anthophyllite [CAS #17068-78-9], chrysotile [CAS #12001-29-5], crocidolite [CAS #12001-28-4], tremolite [CAS #14567-73-8]); fibrous glass.

SAMPLE INFO	MEASUREMENT
SAMPLER: FILTER (0.8-1.2 $\mu\text{m}$ cellulose ester membrane, 25-mm diameter)	!TECHNIQUE: MICROSCOPY, PHASE CONTRAST ! !ANALYTE: fibers (manual count) ! !SAMPLE PREPARATION: acetone/triacetin method ! !COUNTING RULES: Set A (P&CAM 239 [3,4]) or Set B (modified CRS [5]) ! !EQUIPMENT: 1. phase-contrast microscope 2. Walton-Beckett graticule (100 $\mu\text{m}$ field diameter): A Rules use G-22; B Rules use Type G-24 3. phase-shift test slide (HSE/NPL) ! !CALIBRATION: phase-shift detection limit about 3 degrees [7] ! !RANGE: 100 to 1300 fibers/mm <sup>2</sup> filter area [6] ! !ESTIMATED LOD: 7 fibers/mm <sup>2</sup> filter area ! !PRECISION: 0.10 to 0.12 [3] (A Rules) !
FLOW RATE*: $\geq 0.5$ L/min	
VOL-MIN*: 400 L @ 0.1 fiber/mL -MAX*: 1920 L @ 0.1 fiber/mL *Adjust for 100 to 1300 fibers/mm <sup>2</sup> (step 4)	
SHIPMENT: routine	
SAMPLE STABILITY: indefinite	
BLANKS: 10% of samples (minimum 2) [3]	
ACCURACY	
RANGE STUDIED: 80 to 100 fibers counted	
BIAS: see EVALUATION OF METHOD	
OVERALL PRECISION ( $s_p$ ): 0.115 to 0.13 [3] (A Rules)	

APPLICABILITY: The working range is 0.02 fiber/mL (1920-L air sample) to 1.25 fibers/mL (400-L air sample). The method gives an index of airborne asbestos fibers but may be used for other materials such as fibrous glass by inserting suitable parameters into the counting rules. The method does not differentiate between asbestos and other fibers. Asbestos fibers less than ca. 0.25  $\mu\text{m}$  diameter will not be detected by this method [7].

INTERFERENCES: Any other airborne fiber may interfere since all particles meeting the counting criteria are counted. Chain-like particles may appear fibrous. High levels of non-fibrous dust particles may obscure fibers in the field of view and raise the detection limit.

OTHER METHODS: This method introduces changes for improved sensitivity and reproducibility and replaces P&CAM 239 [3,4].

2/15/84

7400-1

000091

## REAGENTS:

1. Acetone.\*
2. Triacetin (glycerol triacetate), reagent grade.

\*See Special Precautions.

## EQUIPMENT:

1. Sampler: field monitor, 25 mm, three-piece cassette with 50-mm extension cowl with cellulose ester filter, 0.8 to 1.2- $\mu$ m pore size and backup pad.  
NOTE: Analyze representative filters for fiber background before use and discard the filter lot if more than 5 fibers/100 fields are found.
2. Personal sampling pump,  $\geq 0.5$  L/min (see step 4 for flow rate), with flexible connecting tubing.
3. Microscope, phase contrast, with green or blue filter, 8 to 10X eyepiece, and 40 to 45X phase objective (total magnification ca. 400X); numerical aperture = 0.65 to 0.75.
4. Slides, glass, single-frosted, pre-cleaned, 25 x 75 mm.
5. Cover slips, 25 x 25 mm, no. 1-1/2, unless otherwise specified by microscope manufacturer.
6. Knife, #10 surgical steel, curved blade.
7. Tweezers.
8. Flask, Luth-type, insulated neck, 250 to 500 mL (with single-holed rubber stopper and elbow-jointed glass tubing, 16 to 22 cm long).
9. Hotplate, spark-free, stirring type; heating mantle; or infrared lamp and magnetic stirrer.
10. Syringe, hypodermic, with 22-gauge needle.
11. Graticule, Walton-Beckett type with 100  $\mu$ m diameter circular field at the specimen plane (area = 0.00785 mm<sup>2</sup>) (Type G-22 for A Rules; Type G-24 for B Rules). Available from Graticules Ltd., Morley Road, Tonbridge TN9 1RN, Kent, England (Telephone 011-44-732-359061).  
NOTE: The graticule is custom-made for each microscope. Specify disc diameter needed to fit exactly the ocular of the microscope and the diameter (mm) of the circular counting area (see step 11).
12. HSE/NPL phase contrast test slide, Mark II. Available from PTR Optics Ltd., 145 Newton Street, Waltham, MA 02154 (Telephone (617) 891-6000).
13. Telescope, ocular phase-ring centering.
14. Stage micrometer (0.01 mm divisions).

**SPECIAL PRECAUTIONS:** Acetone is an extremely flammable liquid and precautions must be taken not to ignite it. Heating of acetone must be done in a ventilated laboratory fume hood using a flameless, spark-free heat source.

## • SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line [3].
2. Fasten the sampler to the worker's lapel as close as possible to the worker's mouth.  
Remove the top cover from the end of the cowl extension (open face) and orient face down.  
Wrap the joint between the extender and monitor body with shrink tape to prevent air leaks.



3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove the caps from the field blank cassettes and store the caps and cassettes in a clean area (bag or box) during the sampling period. Replace the caps in the cassettes when sampling is completed.
4. Sample at 0.5 L/min or greater [8]. Do not exceed the dust loading on the filter. Adjust sampling flow rate,  $Q$  (L/min), and time to produce a dust density,  $E$  (fibers/mm<sup>2</sup>), of 100 to 1300 fibers/mm<sup>2</sup> [ $3.85 \times 10^4$  to  $5 \times 10^5$  fibers per 25-mm filter with effective collection area ( $A_c = 385$  mm<sup>2</sup>)] for optimum counting precision (see step 21). Calculate the minimum sampling time,  $t_{min}$  (min), at the action level (one-half the current standard),  $L$  (fibers/mL), of the fibrous aerosol being sampled:

$$t_{min} = \frac{(A_c)(E)}{(Q)(L)10^3}$$

5. Remove the field monitor at the end of sampling, replace the plastic top cover and small end caps, and store the monitor.
  6. Ship the samples in a rigid container with sufficient packing material to prevent jostling or damage.
- NOTE: Do not use polystyrene foam in the shipping container because of electrostatic forces which may cause fiber loss from the sampler filter.

#### SAMPLE PREPARATION:

NOTE: The object is to produce samples with a smooth (non-grainy) background in a medium with a refractive index equal to or less than 1.46. The method below collapses the filter for easier focusing and produces permanent mounts which are useful for quality control and interlaboratory comparison. Other mounting techniques meeting the above criteria may also be used (e.g., the non-permanent field mounting technique used in P&CAM 239 [1,3,4]).

7. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Place 40 to 60 mL of acetone into a Guth-type flask. Stopper the flask with a single-hole rubber stopper through which a glass tube extends 5 to 8 cm into the flask. The portion of the glass tube which exits the top of the stopper (8 to 10 cm) is bent downward in an elbow which makes an angle of 20 to 30° with the horizontal.
9. Place the flask on a stirring hotplate or wrap in a heating mantle. Heat the acetone gradually to its boiling temperature (ca. 58 °C).

CAUTION: The acetone vapor must be generated in a ventilated fume hood away from all open flames and spark sources. Alternate heating methods can be used, providing no open flame or sparks are present.

10. Mount either the whole sample filter or a wedge cut from the sample filter on a clean glass slide.
  - a. Cut wedges of ca. 25% of the filter area with a curved blade steel surgical knife using a rocking motion to prevent tearing.
  - b. Place the filter or wedge, dust side up, on the slide. Static electricity will usually keep the filter on the slide until it is cleared.
  - c. Hold the glass slide supporting the filter approximately 1 to 2 cm from the glass tube port where the acetone vapor is escaping from the heated flask. The acetone vapor stream should cause a condensation spot on the glass slide ca. 2 to 3 cm in diameter. Move the glass slide gently in the vapor stream. The filter should clear in 2 to 5 sec. If the filter curls, distorts or is otherwise rendered unusable, the vapor stream is probably not strong enough. Periodically wipe the outlet port with tissue to prevent liquid acetone dripping onto the filter.



- d. Using the hypodermic syringe with a 22-gauge needle, place 1 to 2 drops of triacetin on the filter. Gently lower a clean 25-mm square cover slip down onto the filter at a slight angle to reduce the possibility of forming bubbles. If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours.
  - e. Glue the edges of the cover slip to the glass slide using a lacquer or nail polish [9].
- NOTE: If clearing is slow, the slide preparation may be heated on a hotplate (surface temperature 50 °C) for 15 min to hasten clearing. Counting may proceed immediately after clearing and mounting are completed.

CALIBRATION AND QUALITY CONTROL:

11. Calibration of the Walton-Beckett graticule. The diameter,  $d_c$  (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.
  - a. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
  - b. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.
  - c. Install the 40 to 45X phase objective.
  - d. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
  - e. Measure the magnified grid length,  $L_0$  ( $\mu$ m), using the stage micrometer.
  - f. Remove the graticule from the microscope and measure its actual grid length,  $L_a$  (mm). This can best be accomplished by using a stage fitted with verniers.
  - g. Calculate the circle diameter,  $d_c$  (mm), for the Walton-Beckett graticule:

$$d_c = \frac{L_a}{L_0} \times D.$$

Example: If  $L_0 = 108 \mu$ m,  $L_a = 2.93$  mm and  $D = 100 \mu$ m, then  $d_c = 2.71$  mm.

- h. Check the field diameter,  $D$  (acceptable range  $100 \mu$ m  $\pm$  2  $\mu$ m) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area ( $\text{mm}^2$ ).
12. Microscope adjustments. Follow the manufacturer's instructions and also the following:
  - a. Adjust the light source for even illumination across the field of view at the condenser iris.

NOTE: Köhler illumination is preferred, where available.

  - b. Focus on the particulate material to be examined.
  - c. Make sure that the field iris is in focus, centered on the sample and open only enough to fully illuminate the field of view.
  - d. Use the telescope ocular supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric.
13. Check the phase-shift detection limit of the microscope periodically.
  - a. Remove the HSE/NPL phase-contrast test slide from its shipping container and center it under the phase objective.
  - b. Bring the blocks of grooved lines into focus.

NOTE: The slide consists of seven sets of grooves (ca. 20 grooves to each block) in descending order of visibility from sets 1 to 7. The requirements for asbestos counting are that the microscope optics must resolve the grooved lines in set 3 completely, although they may appear somewhat faint, and that the grooved lines in sets 6 and 7 must be invisible. Sets 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has either too low or too high a resolution to be used for asbestos counting.

- c. If the image quality deteriorates, clean the microscope optics and if the problem persists, consult the microscope manufacturer.
14. Quality control of fiber counts
  - a. Prepare and count field blanks along with the field samples. Report the counts on each blank. Calculate the mean of the field blank counts and subtract this value from each sample count before reporting the results.

NOTE 1: The identity of the blank filters should be unknown to the counter until all counts have been completed.

NOTE 2: If a field blank yields fiber counts greater than 7 fibers/100 fields, report possible contamination of the samples.
  - b. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter).
15. Use the following test to determine whether a pair of counts on the same filter should be rejected because of possible bias. This statistic estimates the counting repeatability at the 95% confidence level. Discard the sample if the difference between the two counts exceeds  $2.77 (F) s_r$ , where  $F$  = average of the two fiber counts and  $s_r$  = relative standard deviation, which should be derived by each laboratory based on historical in-house data.

NOTE: If a pair of counts is rejected as a result of this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts.
16. Enroll each new counter in a training course which compares performance of counters on a variety of samples using this procedure.

NOTE: To ensure good reproducibility, all laboratories engaged in asbestos counting should participate in an asbestos proficiency testing program such as the NIOSH Proficiency Analytical Testing (PAT) Program and routinely participate with other asbestos fiber counting laboratories in the exchange of field samples to compare performance of counters.

## MEASUREMENT:

17. Place the slide on the mechanical stage of the calibrated microscope with the center of the filter under the objective lens. Focus the microscope on the plane of the filter.
18. Regularly check phase-ring alignment and Köhler illumination [7].
19. Select one of the following sets of counting rules:

NOTE: The two sets of rules have been demonstrated to produce equivalent mean counts on a variety of asbestos sample types [5] and must be strictly followed in order to obtain valid results. No hybridizing of the two sets of rules is permitted. The calibration of the microscope with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25  $\mu$ m).

  - a. A Rules (same as P&CAM 239 rules [1,3,4]).

NOTE: The A Rules are required for monitoring asbestos for compliance purposes under OSHA or NIOSH standards.

    1. Count only fibers longer than 5  $\mu$ m. Measure the length of curved fibers along the curve.
    2. Count only fibers with a length-to-width ratio equal to or greater than 3:1.
    3. For fibers which cross the boundary of the graticule field, do the following:
      - a. Count any fiber longer than 5  $\mu$ m which lies entirely within the graticule area.
      - b. Count as 1/2 fiber any fiber with only one end lying within the graticule area.
      - c. Do not count any fiber which crosses the graticule boundary more than once.
      - d. Reject and do not count all other fibers.



4. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
5. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 fields regardless of fiber count.

#### B. B Rules

NOTE: The B Rules are preferred analytically because of their demonstrated ability to improve the reproducibility of fiber counts [5].

1. Count only ends of fibers. Each fiber must be longer than 5  $\mu\text{m}$  and less than 3  $\mu\text{m}$  diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules b.1 and b.2.
4. Count visibly free ends which meet rules b.1 and b.2 when the fiber appears to be attached to another particle, regardless of the size of the other particle.
5. Count the free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules b.1 and b.2.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 fields. Stop at 100 fields, regardless of the fiber count.
7. Divide the total end count by 2 to yield fiber count.

NOTE: Split fibers will normally be counted as more than two ends if the free ends meet the rules b.1. and b.2.

20. Start counting from one end of the filter and progress along a radial line to the other end, shift either up or down on the filter and continue in the reverse direction [10]. Select fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. When an agglomerate covers ca. 1/6 or more of the field of view, reject the field and select another. Do not report rejected fields in the number of total fields counted.

NOTE: When counting a field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count.

#### CALCULATIONS:

21. Calculate and report fiber density on the filter,  $E$  (fibers/ $\text{mm}^2$ ), by dividing the total fiber count,  $F$ , minus the mean field blank count,  $B$ , by the number of fields,  $n$ , and the field area,  $A_f$  (0.00785  $\text{mm}^2$  for a properly calibrated Walton-Beckett graticule):

$$E = \frac{(F - B)}{(n)(A_f)} \text{ fibers/mm}^2.$$

22. Calculate the concentration,  $C$  (fibers/mL), of fibers in the air volume sampled,  $V$  (L), using the effective collection area of the filter,  $A_c$  (385  $\text{mm}^2$  for a 25-mm filter):

$$C = \frac{(E)(A_c)}{V \cdot 10^3}$$

\* NOTE: Periodically check and adjust the value of  $A_c$ , if necessary.

#### EVALUATION OF METHOD:

This method is a revision of NIOSH Method P&CAM 239 [1,3,4]. A summary of the revisions is as follows:



## A. Sampling

The change from a 37-mm to a 25-mm filter size was incorporated to improve sensitivity and reduce problems associated with non-uniform fiber loading reported on the 37-mm filters [10]. The change in flow rates allows for 2 m<sup>3</sup> full-shift samples to be taken, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not affected by changes in flow rate in the range 0.5 to 16 L/min [5].

## B. Sample Preparation Technique

The acetone vapor-triacetin preparation technique has been incorporated in the method as a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [1,3,4,11].

## C. Measurement

1. The inclusion of the Walton-Beckett graticule in the method was made to standardize the field area observed through the eyepiece [6,11].
2. The introduction of the HSE/NPL test slide was made to standardize microscope optics for sensitivity to fiber diameter [7,11].
3. A recent international collaborative study involved 16 laboratories using prepared slides from the asbestos, cement, milling, mining, textile, and friction material industries [5]. The relative levels of count by different counting rules were:

Sample Type	Number of Samples	Aspect Ratio > 3:1		Aspect Ratio > 5:1	
		AIA	Mod. CRS*	AIA	Mod. CRS*
Mining	10	100	127	74	92
Milling	10	100	112	84	95
Asbestos Cement	14	100	146	90	137
Textile Chrysotile	10	100	109	89	99
Friction Material	10	100	130	87	116
Others (Insulation, Amosite)	6	100	127	92	118
TOTAL: 60		MEAN: 100	125	86	110

\*Arithmetic means of counts made by different laboratories relative to the AIA counts.

The modified CRS (NIOSH B) Rules were found to be more precise than the AIA (NIOSH A)\* Rules. The ranges of relative standard deviations ( $s_r$ ) which varied with sample type and laboratory were:

	$s_r$		
	Intralaboratory	Interlaboratory	Overall
AIA (NIOSH A Rules)*	0.12 to 0.40	0.27 to 0.85	0.46
Modified CRS (NIOSH B Rules)	0.11 to 0.29	0.20 to 0.35	0.25

\*Under AIA rules, only fibers having a diameter less than 3  $\mu$ m are counted and fibers attached to particles larger than 3  $\mu$ m are not counted. NIOSH A Rules are otherwise similar to the AIA rules.

The B Rules have also been favorably received by analysts as less ambiguous and simpler to use; these rules also showed the least bias relative to AIA rules in the collaborative study. An independent NIOSH laboratory study using amosite fibers reported a relative standard deviation, including within- and between-sample

variability, of 0.157 for the B Rules [12]. Adding an estimated sampling pump error,  $s_p$ , of 0.05 [13] to the within-sample variability in this study results in an estimate of overall precision,  $s_r$ , of 0.102 for the B Rules.

4. Because of past inaccuracies associated with low fiber counts, the minimum loading has been increased to 100 fibers/mm<sup>2</sup> filter area (80 fibers total count). This level yields an overall  $s_r = 0.13$ , as indicated in Figure 3 (revised) of P&CAM 239 [3,4] which corresponds to a measurement  $s_r = 0.12$  after removal of pump error [13]. Similarly, at the maximum count of 100 fibers, overall  $s_r = 0.115$  and measurement  $s_r = 0.10$  are obtained.

D. Evaluation of the method using the A and B counting rules will proceed on a continuing basis through the NIOSH Proficiency Analytical Testing (PAT) Program. The new PAT reporting form allows for reporting of results by either set of rules as of January, 1984.

#### REFERENCES:

- [1] Revised Recommended Asbestos Standard, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-169 (1976).
- [2] Criteria for a Recommended Standard...Occupational Exposure to Fibrous Glass, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-152 (1977).
- [3] Leidel, N. A., S. G. Bayer, R. D. Zumwalde, and K. A. Busch. USPHS/NIOSH Membrane Filter Method for Evaluating Airborne Asbestos Fibers, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-127 (1979).
- [4] NIOSH Manual of Analytical Methods, 2nd. ed., V. 1., P&CAM 239, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
- [5] Crawford, N. P., H. L. Thorpe, and W. Alexander. "A Comparison of the Effects of Different Counting Rules and Aspect Ratios on the Level and Reproducibility of Asbestos Fiber Counts," Part I: Effects on Level (Report No. TM/82/23), Part II: Effects on Reproducibility (Report No. TM/82/24), Institute of Occupational Medicine, Edinburgh, Scotland (December, 1982).
- [6] Walton, W. H. "The Nature, Hazards, and Assessment of Occupational Exposure to Airborne Asbestos Dust: A Review," *Ann. Occup. Hyg.*, **25**, 115-247 (1982).
- [7] Rooker, S. J., N. P. Vaughn, and J. M. LeGuen. "On the Visibility of Fibers by Phase Contrast Microscopy," *Amer. Ind. Hyg. Assoc. J.*, **43**, 505-515 (1982).
- [8] Johnston, A. M., A. D. Jones, and J. H. Vincent. "The Influence of External Aerodynamic Factors on the Measurement of the Airborne Concentration of Asbestos Fibres by the Membrane Filter Method," *Ann. Occup. Hyg.*, **25**, 309-316 (1982).
- [9] Asbestos International Association, AIA Health and Safety Recommended Technical Method #1 (RTH1). "Airborne Asbestos Fiber Concentrations at Workplaces by Light Microscopy" (Membrane Filter Method), London (1979).
- [10] Hooke, M. B., C. E. Feigley, and D. A. Ludwig. "Interwedge Variation in the Membrane Filter Method for Airborne Asbestos Fibers," *Amer. Ind. Hyg. Assoc. J.*, **44**, 542-546 (1983).
- [11] Chatfield, E. J. Measurement of Asbestos Fibre Concentrations in Workplace Atmospheres, Royal Commission on Matters of Health and Safety Arising from the Use of Asbestos in Ontario, Study No. 9, 180 Dundas Street West, 22nd Floor, Toronto, Ontario, CANADA M5G 1Z8.
- [12] Taylor, D. G., P. A. Baron, S. A. Shulman and J. W. Carter. "Identification and Counting of Asbestos Fibers," *Am. Ind. Hyg. Assoc. J.*, **45**(2), 84-88 (1984).
- [13] Busch, K. A. and D. G. Taylor. "Statistical Protocol for the NIOSH Validation tests", Chemical Hazards in the Workplace, Measurement and Control, ACS Symposium Series 149, American Chemical Society, Washington, DC (1981).

METHOD REVISED BY: James W. Carter, David G. Taylor, Ph.D., CIH, and Paul A. Baron, Ph.D., NIOSH/DPSE; based on the revised Method P&CAM 239 [1,3,4].



IDENTIFICATION AND QUANTIFICATION OF ASEESTIFORM  
MINERALS IN BULK INSULATION SAMPLES by  
POLARIZED LIGHT MICROSCOPY.

INDUSTRIAL HYGIENE DEPARTMENT

Date: September 8, 198

SUMMARY OF METHOD

Examination of bulk samples by polarized light microscopy enables the microscopist to identify the minerals present in the sample and estimate their percentages. The identification process is based on the morphology and optical properties of the minerals. The quantification is based on the area occupied by each of the minerals.

1. Apparatus

1.1 A low power binocular microscope, preferably stereoscopic, shall be used to examine the bulk insulation sample as received.

1.1.1 Microscope: binocular, 10-20X.

1.1.2 Light Source: incandescent or fluorescent.

1.1.3 Forceps, Dissecting Needles, and Probes

1.1.4 Glassine Paper or Clean Glass Plate: 4" x 4".

1.2 Sample preparation apparatus requirements will depend upon the insulation sample type under consideration. Various physical and/or chemical means must be employed for an adequate sample assessment.

1.2.1 Mortar and Pestle: agate or porcelain.

1.2.2 Wiley Mill - High Speed Cutting Machine

1.2.3 Razor Blade

1.3 Compound microscope requirements: A polarized light microscope complete with polarizer, analyzer wave plate retardation port, graduated rotating stage, substage condenser, lamp, and lamp iris.

1.3.1 Polarized Light Microscope: described above.

1.3.2 Objective Lenses: 10X and 45X or near equivalent.

1.3.3 Objective Lens Dispersion Staining (optional)

000099



- 1.3.4 Ocular Lens: 10X minimum.
- 1.3.5 Eyeiece Reticule: squared grid-indexed, 5 mm scale length.
- 1.3.6 Stage Micrometer
- 1.3.7 Michel Levy Interference Color Chart (optional)
- 1.3.8 Microscope Slides
- 1.3.9 Coverslips: No. 1.0-1.5, 18 mm diameter
- 1.3.10 Interferometer (optional)
- 1.3.11 Gypsum Wave Retardation Plate

## 2. Reagents

### 2.1 Sample Preparation

- 2.1.1 10% HCl ACS reagent grade

### 2.2 Analytical Reagents

- 2.2.1 Refractive Index Oils: 1.500-1.750 in 0.002-step increments.
  - 2.2.2 Refractive Index Oils: high dispersion, 1.550, 1.605, 1.630, 1.680, and 1.700 (optional)
  - 2.2.3 UICC Asbestos Reference Sample Set
  - 2.2.4 Asbestiform Tremolite
  - 2.2.4 Asbestiform Actinolite
- 2.3 Permanent Slide Standards - a known amount of asbestos mixed with other constituents.

## 3. Procedures

- 3.1 Sampling: Samples for analysis of asbestos content shall be taken in the manner prescribed in the guidance document Asbestos-Containing Materials in School Buildings, EPA No. C00090, part 1. If there are any questions about the representative nature of the sample, another sample should be requested before proceeding with the analysis.

000100

## 3.2 Analysis

- 3.2.1 Gross Examination: Bulk samples of insulation materials taken for the identification and quantification of asbestos are first examined for homogeneity with the aid of a low magnification dissecting microscope. The core sample is carefully removed from the sampling container onto a glassine transfer paper or clean glass plate and note is made of the top and bottom orientation. The sample is examined under low power magnification for layering. When discrete strata are identified, each is treated as a separate material so that fibers are identified and quantitated in that layer and then as a portion of the whole sample. Subsamples of each material type observed with low power magnification from the several layers will be prepared separately for examination with polarized light microscopy.
- 3.2.2 Matrix Reduction: When subsamples of the material types viewed above are not readily obtainable by simple selection means, a matrix reduction step must be employed to access the material before further characterization. The selection of procedure is dependent upon the samples encountered and personal preference. A clean mortar and pestle can be used effectively in size reduction of soft or loosely bound materials. Periodic checks of the particle sizes should be made during the grinding operation so as to preserve any fiber bundles present in an identifiable form. The Wiley Mill should be used to homogenize fibrous material, which is usually hard to grind by mortar and pestle.
- 3.2.3 For positive identification of asbestos, it is expected that results from the following six parameters will be within the range of or consistent with published values (See Flow-Chart).
- Morphology - size, shape, aspect ration, etc. (Table I).
  - Color and pleochroism (Table I).
  - Refractive indices (Table I).
  - Birefringence (Table I).
  - Extinction characteristics (Table I).
  - Sign of elongation (Table I).
- Certain of the above parameters may vary with the mineral source and the sample's previous history of treatment, such as acid contact or heating. Heat, for example, can change the color and refractive

000101

index of all the asbestiform minerals. When fibers of forsterite or other thermal decomposition products of asbestos are identified, this fact should be noted clearly in the report with a request for additional sample from another location. Confirmation for positive identification should be performed routinely by dispersion staining (Table II).

- 3.2.4. Quantitation of Asbestos Content: Asbestos quantitation may be performed in several different ways, again according to the sample type under examination. For homogeneous-appearing materials, portions of the whole sample may be taken from the sample mass in ten different places with a fine forceps. Take precautions to limit stringing out of additional fibrous material in this process. The collection so obtained is mixed and then spread over multiple slides.

Determination of asbestos content as an areal entity will be done by systemic scanning of the slides and comparing them to the permanent slide standards.

4. Range

The range of the analysis is dependent on the amount of material examined. Minimal specifications for reporting results are set at 1.0 percent for detection of asbestiform minerals by polarized light microscopy. Lesser quantities of asbestos in a mixed insulation material sample will be subject to wider variation in reported results because of sampling variation in an inhomogeneous matrix. Upper limits are unbounded. There is no measure of sensitivity presently available.

5. Interferences

Other fibrous organic and inorganic constituents may pose a challenge to identification, separation, and quantitation of the asbestiform mineral content from nonasbestos materials. Spray-on binder materials may coat fibers to impart color and change other optically determined parameters to the extent of masking the fiber identity. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification.

6. Precision and Accuracy

Adequate data for accuracy and precision measurements are not currently available.

7. References

- Optical Mineralogy, John F. Kerr, McGraw Hill, New York, N.Y.
- The Particle Atlas, Edition 2, W. C. McGrone, Ann Arbor Science Publishers.

000102



7. References (continued)

- NBS Special Publication 500, Workshop on Asbestos: Definitions and Measurement Methods. Edited by C. Gravatt, P.D. LaFleur, and R.F.J. Heanrich. 1978.
- Selected Silicate Minerals and Their Asbestiform Varieties, Mineralogical Definitions, and Identification Characteristics, USBM Information Circular 8751, W. J. Campbell, R. L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg, 1977.
- Polarized Light Microscopy, W.C. McCrone, L.B. McCrone, and J.G. Delly, Ann Arbor Science Press, 1978.
- Asbestos-Containing Materials in School Buildings: A Guidance Document, EPA/OTS No. C00090, 1979.
- Identification of Asbestos Fibers and Other Fibers and Minerals in Bulk Samples by Polarized Light Microscopy: Training Procedure. Y. Yaffe, LFE Corp., Environmental Analysis Laboratories.
- Tentative Method For The Determination of Asbestiform Minerals In Bulk Insulation Samples By Polarized Light Microscopy. U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Research Triangle Park, April 1980.

000153

TABLE I.

Optical Properties of Asbestos Fibers and Other Minerals

Mineral	Max. Extinction Angle	$\alpha$	Indices $\beta$	$\gamma$	Birefringence	Optical Sign	Elongation Sign	
Actinolite	10-15°	1.614	1.630	1.641	0.022-0.027	-		Asbestiform Long prismatic crystals and columnar to fibrous
Amosite	0°	1.695		1.700	0.025-0.027	+	+	Asbestiform Straight long fibers
Anthophyllite	0°	1.598- 1.652	1.630- 1.655	1.623 1.676	0.016-0.025	+	+	Asbestiform Long prismatic crystals and columnar to fibrous
Chrysotile	0°	1.493- 1.546	1.504- 1.550	1.517- 1.557	0.011-0.014	+	+	Asbestiform Cross-fiber pin-lets
Crocidolite	0°-3°	1.680- 1.698	1.683- 1.700	1.685- 1.706	0.004	-	-	Asbestiform (pleochroic) Long delicate blue-green fibers
Cummingtonite	15-20°	1.639- 1.657	1.645- 1.669	1.664- 1.686	0.027	+	+	Usually in aggregates of prismatic crystals
Grunerite	10-15°	1.657- 1.663	1.684- 1.697	1.699- 1.717	0.042-0.054	-	+	Asbestiform Usually in columnar aggregates
Lizardite	0°	1.538- 1.546		1.546- 1.560	0.015	-	+	Fine grained

000104

TABLE 1.

Optical Properties of Asbestos Fibers and Other Minerals

Mineral	Max. Extinction Angle	$\alpha$	Indices $\beta$ $\gamma$		Birefringence	Optical Sign	Elongation Sign	
Talc	0-3°	1.538- 1.545	1.575- 1.590	1.575- 1.590	0.030-0.05	-	+	Coarse to fine platy or fibrous aggregates
Tremolite	10-20°	1.600- 1.628	1.613- 1.644	1.625- 1.655	0.022-0.027	-	+	Asbestiform Long prismatic columnar to fibrous aggregates
Antigorite	0°	1.555- 1.564	1.562- 1.573	1.562- 1.573	0.008	-	+	Anhydral crystal or aggregates of fibro-lamellar structure
<u>MICA</u>								
Biotite		1.541- 1.579	1.574- 1.638	1.579- 1.638	0.040-0.060	-		Equihedral six-sided crystals that are usually tabular
Muscovite		1.556- 1.570	1.587- 1.607	1.593- 1.611	0.037-0.041	-		Thin tabular crystals or small aggregates
Vermiculite		1.525- 1.561		1.542- 1.581	0.020-0.031	-		Thin tabular crystals. Trans- parent to opaque Colorless to light pleochroic

000105



TABLE 1.

Optical Properties of Asbestos Fibers and Other Minerals

Mineral	Max. Extinction Angle	Indices		Birefringence	Optical Sign	Elongation Sign
<u>CALCITE GROUP</u>						
1. Calcite		1.486	1.658	0.172	-	Fine to coarse aggregates. Rhombohedral cleavage.
2. Dolomite		1.500- 1.526	1.680- 1.716	0.180	-	Fine to coarse grained & rhombohedral crystal of rhombohedral
3. Magnesite		1.509- 1.527	1.700- 1.726	0.191-0.199	-	Fine to coarse aggregates
Quartz		1.553	1.544	0.009	+	Sharply pointed fragments, often show conchoidal fracture.

000106

TABLE 1.

Optical Properties of Asbestos Fibers and Other Minerals

Mineral	Max. Extinction Angle	$\alpha$	Indices $\beta$	$\gamma$	Birefringence	Optical Sign	Elongation Sign
Gypsum		1.520	1.522	1.529	0.009	+	
Orthoclase							

Due to coarse  
grained. Some  
times fiber  
structure

000107

TABLE II

Identification of Asbestos Fibers and Other Minerals by Dispersion Staining

Type of Mineral	Cargille Liquid $n_D^{25^\circ}$ for Mounting	Appearance (colors seen)					
		With Polarizer				No Polarizer	
		Central Stop Fiber Orientation		Annular Stop		Central Stop	Annular Stop
		EW	NS	EW	NS		
Actinolite							
Amosite	1.670	Green-blue	Yellow-orange	Pale Yellow	Dark blue	brown-yellow	dark blue or yellow
	1.700	Pink-blue	Turquoise	Yellow-orange	pale yellow	turquoise	yellow-orange
Anthophyllite	1.606	turquoise mainly	brown-red	hardly seen	hardly seen	pale orange yellow	hardly seen
	1.670	yellow-green	yellow green	green-blue	magenta	green blue	pale yellow
Chrysotile	1.560	turquoise	dark blue	orange-red	yellow-orange	turquoise	orange
Crocidolite	1.700	blue-green	blue-green	yellow	dark blue	turquoise	yellow blue
	1.670	brown-yellow	brown-yellow	natural blue	natural blue	brown yellow	natural blue
Tremolite	1.606	turquoise	golden yellow	yellow orange	Dark blue	yellow orange	dark blue
	1.670	yellow green	light turquoise	yellow magenta green	yellow-green	yellow - pale green	pale yellow yellow-magenta green
Wollastonite	1.670	yellow-green	green-yellow	yellow magenta	yellow-magenta	turquoise-blue	yellow-brown
	1.606	orange-brown	yellow-orange	dark blue	dark blue	yellow orange	dark blue

000108



TABLE I.

## Optical Characteristics of Plant and Man-Made Fibers (The Particle Atlas, Vol. II)

Mineral	Max. Extinction Angle	Indices Length- wise	Cross- wise	Birefringence	Elongation	Comments
Jute	0°	1.517	1.536	0.041		transparent, colorless, usually straight cylinders. Thick cell walls mostly in fiber bundles.
Modacrylic	0°	1.535	1.533	0.002	+	Transparent, colorless cylinders
Polyamide (Nylon)	0°	1.580	1.520	0.060		Transparent, colorless cylinders. They may be dyed by variety of color
Polyester (Dacron)	Complete	1.710	1.535	0.175		Transparent, colorless cylinders
Sisal	None	1.49	1.65	0.16		Transparent, colorless to yellowish tan cylinders. Thick cell walls
Viscose Rayon	Complete 0°	1.535- 1.555	1.515 1.535	0.02		Transparent, colorless, striated cylinders
Nonconiferous Wood (paper)	Undulose to perfect	1.53 to	1.58	Low 1st order	+	Transparent, colorless to light yellow. Large baggy cells, irregular fragments
Coniferous Wood (paper)	incomplete 0°	1.53 to	1.58	Low 1st order	+	Transparent, colorless to pale yellow. Ripped & torn fibers, irregular fragments

000109



## Test Method

# Interim Method for the Determination of Asbestos in Bulk Insulation Samples\*

### 1. Polarized Light Microscopy

#### 1.1 Principle and Applicability

Bulk samples of building materials taken for asbestos identification are first examined for homogeneity and preliminary fiber identification at low magnification. Positive identification of suspect fibers is made by analysis of subsamples with the polarized light microscope.

The principles of optical mineralogy are well established.<sup>1,2</sup> A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows the determination of refractive indices along specific crystallographic axes. Morphology and color are also observed. A retardation plate is placed in the polarized light path for determination of the sign of elongation using orthoscopic illumination. Orientation of the two filters such that their vibration planes are perpendicular (crossed polars) allows observation of the birefringence and extinction characteristics of anisotropic particles.

Quantitative analysis involves the use of point counting. Point counting is a standard technique in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting<sup>3</sup> and the interpretation of point count data<sup>3</sup> is available.

\*An interim method is carefully drafted from available source information. This method is still under investigation and therefore is subject to revision.

This method is applicable to all bulk samples of friable insulation materials submitted for identification and quantitation of asbestos components.

#### 1.2 Range

The point counting method may be used for analysis of samples containing from 0 to 100 percent asbestos. The upper detection limit is 100 percent. The lower detection limit is less than 1 percent.

#### 1.3 Interferences

Fibrous organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Spray-on binder materials may coat fibers and affect color or obscure optical characteristics to the extent of masking fiber identity. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification. Procedures that may be used for the removal of interferences are presented in Section 1.7.2.2.

#### 1.4 Precision and Accuracy

Adequate data for measuring the accuracy and precision of the method for samples with various matrices are not currently available. Data obtained for samples containing a single asbestos type in a simple matrix are available in the EPA report *Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method*.<sup>4</sup>

#### 1.5 Apparatus

##### 1.5.1 Sample Analysis

A low-power binocular microscope, preferably stereoscopic, is used to

000110



examine the bulk insulation sample as received

- **Microscope:** binocular, 10-45X (approximate)
- **Light Source:** incandescent or fluorescent
- **Forceps, Dissecting Needles, and Probes**
- **Glassine Paper or Clean Glass Plate**  
Compound microscope requirements. A polarized light microscope complete with polarizer, analyzer, port for wave retardation plate, 360° graduated rotating stage, substage condenser, lamp, and lamp iris
- **Polarized Light Microscope:** described above
- **Objective Lenses:** 10X, 20X, and 40X or near equivalent
- **Dispersion Staining Objective Lens** (optional)
- **Ocular Lens:** 10X minimum
- **Eye-piece Reticle:** cross hair or 25 point Chalkley Point Array
- **Compensator Plate:** 550 milli-micron retardation

#### 1.5.2 Sample Preparation

Sample preparation apparatus requirements will depend upon the type of insulation sample under consideration. Various physical and/or chemical means may be employed for an adequate sample assessment.

- **Ventilated Hood** or negative pressure glove box
- **Microscope Slides**
- **Coverslips**
- **Mortar and Pestle:** agate or porcelain (optional)
- **Wylie Mill** (optional)
- **Beakers & assorted glassware** (optional)
- **Centrifuge** (optional)
- **Filtration apparatus** (optional)
- **Low temperature asher** (optional)

#### 1.6 Reagents

##### 1.6.1 Sample Preparation

- **Distilled Water** (optional)
- **Dilute  $\text{CH}_3\text{COOH}$ :** ACS reagent grade (optional)
- **Dilute  $\text{HCl}$ :** ACS reagent grade (optional)
- **Sodium metaphosphate ( $\text{NaPO}_3$ )** (optional)

##### 1.6.2 Analytical Reagents

- **Refractive Index Liquids:** 1.490-1.570, 1.590-1.720 in increments of 0.002 or 0.004
- **Refractive Index Liquids for Dispersion Staining:** high-dispersion series, 1.550, 1.605, 1.630 (optional)
- **UICC Asbestos Reference Sample Set:** Available from: UICC MRC

Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan CF6 1XW, UK, and commercial distributors

- **Tremolite-asbestos** (source to be determined)
- **Actinolite-asbestos** (source to be determined)

#### 1.7 Procedures

**Note:** Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations should be carried out in a ventilated hood or glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst and contamination of samples by airborne fibers.

##### 1.7.1 Sampling

Samples for analysis of asbestos content shall be taken in the manner prescribed in Reference 5 and information on design of sampling and analysis programs may be found in Reference 6. If there are any questions about the representative nature of the sample, another sample should be requested before proceeding with the analysis.

##### 1.7.2 Analysis

###### 1.7.2.1 Gross Examination

Bulk samples of building materials taken for the identification and quantitation of asbestos are first examined for homogeneity at low magnification with the aid of a stereomicroscope. The core sample may be examined in its container or carefully removed from the container onto a glassine transfer paper or clean glass plate. If possible, note is made of the orientation of top and bottom surfaces. When discrete strata are identified, each is treated as a separate material so that fibers are first identified and quantified in that layer only, and then the results for each layer are combined to yield an estimate of asbestos content for the whole sample.

###### 1.7.2.2 Sample Preparation

Bulk materials submitted for asbestos analysis involve a wide variety of matrix materials. Representative subsamples may not be readily obtainable by simple means in heterogeneous materials, and various steps may be required to alleviate the difficulties encountered. In most cases, however, the best preparation is made by using forceps to sample at several places from the bulk material. Forcep samples are immersed in a refractive index liquid on a microscope slide,

teased apart, covered with a cover glass, and observed with the polarized light microscope

Alternatively, attempts may be made to homogenize the sample or eliminate interferences before further characterization. The selection of appropriate procedures is dependent upon the samples encountered and personal preference. The following are presented as possible sample preparation steps.

A mortar and pestle can sometimes be used in the size reduction of soft or loosely bound materials, though this may cause matting of some samples. Such samples may be reduced in a Wiley mill. Apparatus should be clean and extreme care exercised to avoid cross-contamination of samples. Periodic checks of the particle sizes should be made during the grinding operation so as to preserve any fiber bundles present in an identifiable form. These procedures are not recommended for samples that contain amphibole minerals or vermiculite. Grinding of amphiboles may result in the separation of fiber bundles or the production of cleavage fragments that have aspect ratios greater than 3:1 and will be classified as asbestos fibers. Grinding of vermiculite may also produce fragments with aspect ratios greater than 3:1.

Acid treatment may occasionally be required to eliminate interferences. Calcium carbonate, gypsum, and bassanite (plaster) are frequently present in sprayed or trowelled insulations. These materials may be removed by treatment with warm dilute acetic acid. Warm dilute hydrochloric acid may also be used to remove the above materials. If acid treatment is required, wash the sample at least twice with distilled water, being careful not to lose the particulates during decanting steps. Centrifugation or filtration of the suspension will prevent significant fiber loss. The pore size of the filter should be 0.45 micron or less.

**Caution:** prolonged acid contact with the sample may alter the optical characteristics of chrysotile fibers and should be avoided.

Coatings and binding materials adhering to fiber surfaces may also be removed by treatment with sodium metaphosphate.<sup>7</sup> Add 10 mL of 10 g/L sodium metaphosphate solution to a small (0.1 to 0.5 mL) sample of bulk material in a 15-mL glass centrifuge tube. For approximately 15 seconds each, stir the mixture on a vortex mixer, place in an ultrasonic bath and then shake by hand. Repeat the series.



Collect the dispersed solids by centrifugation at 1000 rpm for 5 minutes. Wash the sample three times by suspending in 10 mL distilled water and recentrifuging. After washing, resuspend the pellet in 5 mL distilled water, place a drop of the suspension on a microscope slide, and dry the slide at 110°C.

In samples with a large portion of cellulosic or other organic fibers, it may be useful to ash part of the sample and examine the residue. Ashing should be performed in a low temperature ashier. Ashing may also be performed in a muffle furnace at temperatures of 500°C or lower. Temperatures of 550°C or higher will cause dehydroxylation of the asbestos minerals, resulting in changes of the refractive index and other key parameters. If a muffle furnace is to be used, the furnace thermostat should be checked and calibrated to ensure that samples will not be heated at temperatures greater than 500°C.

Ashing and acid treatment of samples should not be used as standard procedures. In order to monitor possible changes in fiber characteristics, the material should be viewed microscopically before and after any sample preparation procedure. Use of these procedures on samples to be used for quantitation requires a correction for percent weight loss.

#### 1.7.2.3 Fiber Identification

Positive identification of asbestos requires the determination of the following optical properties.

- Morphology
- Color and pleochroism
- Refractive indices
- Birefringence
- Extinction characteristics
- Sign of elongation

Table 1-1 lists the above properties for commercial asbestos fibers. Figure 1-1 presents a flow diagram of the examination procedure. Natural variations in the conditions under which deposits of asbestiform minerals are formed will produce exceptions to the published values and differences from the UICC standards. The sign of elongation is determined by use of the compensator plate and crossed polars. Refractive indices may be determined by the Becke line test. Alternatively, dispersion staining may be used. Inexperienced operators may find that the dispersion staining technique is more easily learned, and should consult Reference 9 for guidance. Central stop dispersion staining colors are presented in Table

1-2. Available high-dispersion (HD) liquids should be used.

#### 1.7.2.4 Quantitation of Asbestos Content

Asbestos quantitation is performed by a point-counting procedure. An ocular reticle (cross-hair or point array) is used to visually superimpose a point or points on the microscope field of view. Record the number of points positioned directly above each kind of particle or fiber of interest. Score only points directly over asbestos fibers or nonasbestos matrix material. Do not score empty points for the closest particle. If an asbestos fiber and a matrix particle overlap so that a point is superimposed on their visual intersection, a point is scored for both categories. Point counting provides a determination of the area percent asbestos. Reliable conversion of area percent to percent of dry weight is not currently feasible unless the specific gravities and relative volumes of the materials are known.

For the purpose of this method, "asbestos fibers" are defined as having an aspect ratio greater than 3:1 and being positively identified as one of the minerals in Table 1-1.

A total of 400 points superimposed on either asbestos fibers or nonasbestos matrix material must be counted over at least eight different preparations of representative subsamples. Take eight forcp samples and mount each separately with the appropriate refractive index liquid. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25-50 percent empty area within the fields of view. Count 50 nonempty points on each preparation, using either

- A cross-hair reticle and mechanical stage; or
- A reticle with 25 points (Chalkley Point Array) and counting at least 2 randomly selected fields.

For samples with mixtures of isotropic and anisotropic materials present, viewing the sample with slightly uncrossed polars or the addition of the compensator plate to the plane polarized light path will allow simultaneous discrimination of both particle types. Quantitation should be performed at 100X or at the lowest magnification of the polarized light microscope that can effectively distinguish the sample components. Confirmation of the quantitation result by a second analyst on some percentage of analyzed samples should be used as standard quality control procedure.

The percent asbestos is calculated as follows.

$$\% \text{ asbestos} = (a/n) 100\%$$

where

a = number of asbestos counts.  
n = number of nonempty points counted (400).

If a = 0, report "No asbestos detected." If  $0 < a \leq 3$ , report "<1% asbestos."

The value reported should be rounded to the nearest percent.

#### 1.8 References

1. Paul F. Kerr, *Optical Mineralogy*, 4th ed., New York, McGraw-Hill, 1977.
2. E. M. Charnot and C. W. Mason, *Handbook of Chemical Microscopy, Volume One*, 3rd ed., New York, John Wiley & Sons, 1958.
3. F. Chayes, *Petrographic Modal Analysis: An Elementary Statistical Appraisal*, New York: John Wiley & Sons, 1956.
4. E. P. Brantly, Jr., K. W. Gold, L. E. Myers, and D. E. Lentzen, *Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method*, EPA-600/4-82-021, U.S. Environmental Protection Agency, in preparation.
5. U.S. Environmental Protection Agency, *Asbestos-Containing Materials in School Buildings: A Guidance Document, Parts 1 and 2*, EPA/OTS No. C00090, March 1979.
6. D. Lucas, T. Hartwell, and A. V. Rao, *Asbestos-Containing Materials in School Buildings: Guidance for Asbestos Analytical Programs*, EPA-560/13-80-017A, U.S. Environmental Protection Agency, December 1980.
7. D. H. Taylor and J. S. Bloom, Hexametaphosphate pretreatment of insulation samples for identification of fibrous constituents, *Microscope*, 28, 1980.
8. W. J. Campbell, R. L. Blake, L. L. Brown, E. E. Cather, and J. J. Sjöberg, *Selected Silicate Minerals and Their Asbestiform Varieties: Mineralogical Definitions and Identification-Characterization*, U.S. Bureau of Mines Information Circular 8751, 1977.
9. Walter C. McCrone, *Asbestos Particle Atlas*, Ann Arbor: Ann Arbor Science Publishers, June 1980.

Table 1-1. Optical properties of asbestos fibers

Mineral	Morphology, color <sup>1</sup>	Refractive indices <sup>2</sup>		Birefringence	Extinction	Sign of elongation
		$\alpha$	$\gamma$			
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1. Colorless <sup>3</sup> , nonpleochroic.	1.493-1.560	1.517-1.562 <sup>4</sup> (normally 1.556)	.002- .014	to fiber length	+ (length slow)
Amosite (asbestiform grunerite)	Straight, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present.	1.635-1.696	1.655-1.729 <sup>4</sup> (normally 1.696-1.710)	.020-.033	to fiber length	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight, rigid fibers. Thick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color.	1.654-1.701	1.668-1.717 <sup>4</sup> (normally close to 1.700)	.014-.016	to fiber length	- (length fast)
Anthophyllite- asbestos	Straight, single fibers, some larger composite fibers. Anthophyllite cleavage fragments may be present with aspect ratios < 10:1. <sup>4</sup> Colorless to light brown.	1.596-1.652	1.615-1.676 <sup>4</sup>	.019-.024	to fiber length	+ (length slow)
Tremolite- actinolite- asbestos	Tremolite-asbestos may be present as single or composite fibers. Tremolite cleavage fragments may be present as single crystals with aspect ratios < 10:1. <sup>4</sup> Colorless to pale green.	1.599-1.668	1.622-1.688 <sup>4</sup>	.023-.020	Oblique extinction, 10-20° for fragments. Composite fibers show    extinction.	+ (length slow)

<sup>1</sup> From reference 5, colors cited are seen by observation with plane polarized light. <sup>4</sup> Fibers defined as having aspect ratio >3:1.

<sup>2</sup> From references 5 and 8. <sup>5</sup>  $\perp$  to fiber length.

<sup>3</sup> Fibers subjected to heating may be brownish. <sup>6</sup> || to fiber length.

Table 1-2. Central stop dispersion staining colors \*

Mineral	RI Liquid	$\perp$	
Chrysotile	1.550 <sup>ND</sup>	Blue	Blue-magenta
"Amosite"	1.680	Blue-magenta to pale blue	Golden-yellow
	1.550 <sup>ND</sup>	Yellow to white	Yellow to white
Crocidolite <sup>b</sup>	1.700	Red magenta	Blue-magenta
	1.550 <sup>ND</sup>	Yellow to white	Yellow to white
Anthophyllite- asbestos	1.605 <sup>ND</sup>	Blue	Gold to gold-magenta
Tremolite- asbestos	1.605 <sup>NDc</sup>	Pale blue	Yellow
Actinolite- asbestos	1.605 <sup>ND</sup>	Gold-magenta to blue	Gold
	1.630 <sup>NDc</sup>	Magenta	Golden-yellow

\*From reference 9, colors may vary slightly.

<sup>a</sup>Blue absorption color.

<sup>c</sup>Oblique extinction view.



**Polarized light microscopy qualitative analysis:** For each type of material identified by examination of sample at low magnification. Mount specially dispersed sample in 1.550 RI liquid. (If using dispersion staining, mount in 1.550 HD.) View at 100X with both plane polarized light and crossed polars. More than one fiber type may be present.

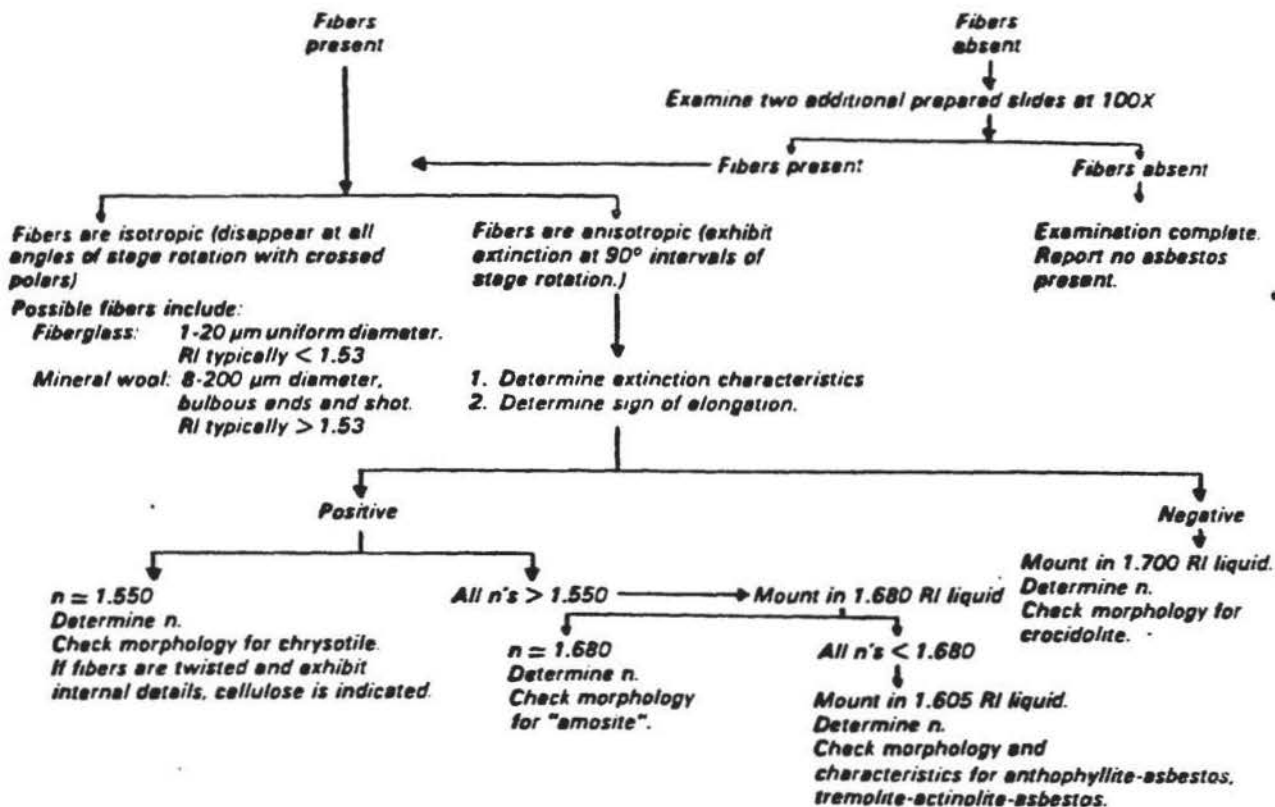


Figure 1-1. Flow chart for qualitative analysis of bulk samples by polarized light microscopy.



## 2. X-Ray Powder Diffraction

### 2.1 Principle and Applicability

The principle of X-ray powder diffraction (XRD) analysis is well established.<sup>1,2</sup> Any solid, crystalline material will diffract an impinging beam of parallel, monochromatic X-rays whenever Bragg's Law,

$$\lambda = 2d \sin \theta,$$

is satisfied for a particular set of planes in the crystal lattice, where

$\lambda$  = the X-ray wavelength, Å;

$d$  = the interplanar spacings of the set of reflecting lattice planes, Å; and

$\theta$  = the angle of incidence between the X-ray beam and the reflecting lattice planes.

By appropriate orientation of a sample relative to the incident X-ray beam, a diffraction pattern can be generated that, in most cases, will be uniquely characteristic of both the chemical composition and structure of the crystalline phases present.

Unlike optical methods of analysis, however, XRD cannot determine crystal morphology. Therefore, in asbestos analysis, XRD does not distinguish between fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-1). However, when used in conjunction with optical methods such as polarized light microscopy (PLM), XRD techniques can provide a reliable analytical method for the identification and characterization of asbestiform minerals in bulk materials.

For *qualitative analysis* by XRD methods, samples are initially scanned over limited diagnostic peak regions for the serpentine (~7.4 Å) and amphibole (8.2-8.5 Å) minerals (Table 2-2). Standard slow-scanning methods for bulk sample analysis may be used for materials shown by PLM to contain significant amounts of asbestos (>5-10 percent). Detection of minor or trace amounts of asbestos may require special sample preparation and step-scanning analysis. All samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals are submitted to a full (5°-60° 2 $\theta$ , 1° 2 $\theta$ /min) qualitative XRD scan, and their diffraction patterns are compared with standard reference powder diffraction patterns<sup>3</sup> to verify initial peak assignments and to identify possible matrix interferences when

subsequent quantitative analysis will be performed.

Accurate *quantitative analysis* of asbestos in bulk samples by XRD is critically dependent on particle size distribution, crystallite size, preferred orientation and matrix absorption effects, and comparability of standard reference and sample materials. The most intense diffraction peak that has been shown to be free from interference by prior qualitative XRD analysis is selected for quantitation of each asbestiform mineral. A "thin-layer" method of analysis<sup>4,5</sup> is recommended in which, subsequent to comminution of the bulk material to ~10  $\mu$ m by suitable cryogenic milling techniques, an accurately known amount of the sample is deposited on a silver membrane filter. The mass of asbestiform material is determined by measuring the integrated area of the selected diffraction peak using a step-scanning mode, correcting for matrix absorption effects, and comparing with suitable calibration standards. Alternative "thick-layer" or bulk methods,<sup>7,8</sup> may be used for *semiquantitative analysis*.

This XRD method is applicable as a confirmatory method for identification and quantitation of asbestos in bulk material samples that have undergone prior analysis by PLM or other optical methods.

### 2.2 Range and Sensitivity

The range of the method has not been determined.

The sensitivity of the method has not been determined. It will be variable and dependent upon many factors, including matrix effects (absorption and interferences), diagnostic reflections selected, and their relative intensities.

### 2.3 Limitations

#### 2.3.1 Interferences

Since the fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-1) are indistinguishable by XRD techniques unless special sample preparation techniques and instrumentation are used,<sup>9</sup> the presence of nonasbestiform serpentines and amphiboles in a sample will pose severe interference problems in the identification and quantitative analysis of their asbestiform analogs.

The use of XRD for identification and quantitation of asbestiform minerals in bulk samples may also be limited by the presence of other interfering materials in the sample. For naturally occurring materials the commonly associated asbestos-related mineral interferences can usually be anticipated. However, for fabricated materials the nature of the interferences may vary greatly (Table 2-3) and present more serious problems in identification and quantitation.<sup>10</sup> Potential interferences are summarized in Table 2-4 and include the following:

- *Chlorite* has major peaks at 7.19 Å and 3.58 Å that interfere with both the primary (7.36 Å) and secondary (3.66 Å) peaks for chrysotile. Resolution of the primary peak to give good quantitative results may be possible when a step-scanning mode of operation is employed.
- *Halloysite* has a peak at 3.63 Å that interferes with the secondary (3.66 Å) peak for chrysotile.
- *Keolinite* has a major peak at 7.15 Å that may interfere with the primary peak of chrysotile at 7.36 Å when present at concentrations of >10 percent. However, the secondary chrysotile peak at 3.66 Å may be used for quantitation.
- *Gypsum* has a major peak at 7.5 Å that overlaps the 7.36 Å peak of chrysotile when present as a major sample constituent. This may be removed by careful washing with distilled water, or by heating to 300°C to convert gypsum to plaster of paris.
- *Cellulose* has a broad peak that partially overlaps the secondary (3.66 Å) chrysotile peak.<sup>8</sup>
- Overlap of major diagnostic peaks of the amphibole asbestos minerals, amosite, anthophyllite, crocidolite, and tremolite, at approximately 8.3 Å and 3.1 Å causes mutual interference when these minerals occur in the presence of one another. In some instances adequate resolution may be attained by using step-scanning methods and/or by decreasing the collimator slit width at the X-ray port.
- *Carbonates* may also interfere with quantitative analysis of the amphibole asbestos minerals, amosite,



anthophyllite, crocidolite, and tremolite. Calcium carbonate ( $\text{CaCO}_3$ ) has a peak at 3.035 Å that overlaps major amphibole peaks at approximately 3.1 Å when present in concentrations of >5 percent. Removal of carbonates with a dilute acid wash is possible, however, if present, chrysotile may be partially dissolved by this treatment.<sup>11</sup>

- A major talc peak at 3.12 Å interferes with the primary tremolite peak at this same position and with secondary peaks of crocidolite (3.10 Å), amosite (3.06 Å), and anthophyllite (3.05 Å). In the presence of talc, the major diagnostic peak at approximately 8.3 Å should be used for quantitation of these asbestiform minerals.

The problem of intraspecies and matrix interferences is further aggravated by the variability of the silicate mineral powder diffraction patterns themselves, which often makes definitive identification of the asbestos minerals by comparison with standard reference diffraction patterns difficult. This variability results from alterations in the crystal lattice associated with differences in isomorphous substitution and degree of crystallinity. This is especially true for the amphiboles. These minerals exhibit a wide variety of very similar chemical compositions, with the result being that their diffraction patterns are characterized by having major (110) reflections of the monoclinic amphiboles and (210) reflections of the orthorhombic anthophyllite separated by less than 0.2 Å.<sup>12</sup>

### 2.3.2 Matrix Effects

If a copper X-ray source is used, the presence of iron at high concentrations in a sample will result in significant X-ray fluorescence, leading to loss of peak intensity with increased background intensity and an overall decrease in sensitivity. This situation may be corrected by use of an X-ray source other than copper; however, this is often accompanied both by loss of intensity and by decreased resolution of closely spaced reflections. Alternatively, use of a diffracted beam monochromator will reduce background fluorescent radiation, enabling weaker diffraction peaks to be detected.

X-ray absorption by the sample matrix will result in overall attenuation of the diffracted beam and may seriously interfere with quantitative analysis. Absorption effects may be

minimized by using sufficiently "thin" samples for analysis.<sup>8,13,14</sup> However, unless absorption effects are known to be the same for both samples and standards, appropriate corrections should be made by referencing diagnostic peak areas to an internal standard<sup>7,8</sup> or filter substrate (Ag) peak.<sup>8,9</sup>

### 2.3.3 Particle Size Dependence

Because the intensity of diffracted X-radiation is particle-size dependent, it is essential for accurate quantitative analysis that both sample and standard reference materials have similar particle size distributions. The optimum particle size (i.e., fiber length) range for quantitative analysis of asbestos by XRD has been reported to be 1 to 10 µm.<sup>15</sup> Comparability of sample and standard reference material particle size distributions should be verified by optical microscopy (or another suitable method) prior to analysis.

### 2.3.4 Preferred Orientation Effects

Preferred orientation of asbestiform minerals during sample preparation often poses a serious problem in quantitative analysis by XRD. A number of techniques have been developed for reducing preferred orientation effects in "thick layer" samples.<sup>7,16</sup> For "thin" samples on membrane filters, the preferred orientation effects seem to be both reproducible and favorable to enhancement of the principal diagnostic reflections of asbestos minerals, actually increasing the overall sensitivity of the method.<sup>12,14</sup> However, further investigation into preferred orientation effects in both thin layer and bulk samples is required.

### 2.3.5 Lack of Suitably Characterized Standard Materials

The problem of obtaining and characterizing suitable reference materials for asbestos analysis is clearly recognized. NIOSH has recently directed a major research effort toward the preparation and characterization of analytical reference materials, including asbestos standards;<sup>18,17</sup> however, these are not available in large quantities for routine analysis.

In addition, the problem of ensuring the comparability of standard reference and sample materials, particularly regarding crystallite size, particle size distribution, and degree of crystallinity, has yet to be adequately addressed. For example, Langer et al.<sup>18</sup> have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant

decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be underestimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

### 2.4 Precision and Accuracy

Precision of the method has not been determined.

Accuracy of the method has not been determined.

### 2.5 Apparatus

#### 2.5.1 Sample Preparation

Sample preparation apparatus requirements will depend upon the sample type under consideration and the kind of XRD analysis to be performed.

- *Mortar and Pestle:* Agate or porcelain
- *Razor Blades*
- *Sample Mill:* SPEX, Inc., freezer mill or equivalent
- *Bulk Sample Holders*
- *Silver Membrane Filters:* 25-mm diameter, 0.45-µm pore size. Selas Corp. of America, Filtronics Div., 1957 Pioneer Road, Huntingdon Valley, PA 19006
- *Microscope Slides*
- *Vacuum Filtration Apparatus:* Gelman No. 1107 or equivalent, and side-arm vacuum flask
- *Microbalance*
- *Ultrasonic Bath or Probe:* Model W140, Ultrasonics, Inc., operated at a power density of approximately 0.1 W/mL or equivalent
- *Volumetric Flasks:* 1-L volume
- *Assorted Pipet*
- *Pipet Bulb*
- *Nonserrated Forceps*
- *Polyethylene Wash Bottle*
- *Pyrex Beakers:* 50-mL volume
- *Desiccator*
- *Filter Storage Cassettes*
- *Magnetic Stirring Plate and Bars*
- *Porcelain Crucibles*
- *Muffle Furnace or Low Temperature Asher*

#### 2.5.2 Sample Analysis

Sample analysis requirements include an X-ray diffraction unit, equipped with:

- *Constant Potential Generator; Voltage and mA Stabilizers*
- *Automated Diffractometer with Step-Scanning Mode*
- *Copper Target X-Ray Tube:* High intensity; fine focus, preferably
- *X-Ray Pulse Height Selector*
- *X-Ray Detector* (with high voltage power supply): Scintillation or proportional counter



- **Focusing Graphite Crystal Monochromator;** or **Nickel Filter** (if copper source is used, and iron fluorescence is not a serious problem)
- **Data Output Accessories:**  
Strip Chart Recorder  
Decade Scaler/Timer  
Digital Printer
- **Sample Spinner** (optional)
- **Instrument Calibration Reference Specimen:**  $\alpha$ -quartz reference crystal (Arkansas quartz standard, #180-147-00, Philips Electronics Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430) or equivalent

## 2.6 Reagents

### 2.6.1 Standard Reference Materials

The reference materials listed below are intended to serve as a guide. Every attempt should be made to acquire pure reference materials that are comparable to sample materials being analyzed.

- **Chrysotile:** UICC Canadian, or NIEHS Plastibest. (UICC reference materials available from: UICC, MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF61XW, UK)
- **Crocidolite:** UICC
- **"Amosite":** UICC
- **Anthophyllite-Asbestos:** UICC
- **Tremolite Asbestos:** Wards Natural Science Establishment, Rochester, NY; Cyprus Research Standard, Cyprus Research, 2435 Military Ave., Los Angeles, CA 90064 (washed with dilute HCl to remove small amount of calcite impurity). Indian tremolite, Rajasthan State, India.
- **Actinolite Asbestos:** (Source to be determined).

### 2.6.2 Adhesive

Tape, petroleum jelly, etc. (for attaching silver membrane filters to sample holders).

### 2.6.3 Surfactant

1 Percent aerosol OT aqueous solution or equivalent.

### 2.6.4 Isopropanol

ACS Reagent Grade.

## 2.7 Procedure

### 2.7.1 Sampling

Samples for analysis of asbestos content shall be collected as specified in EPA Guidance Document #CO090, *Asbestos-Containing Materials in School Buildings*.<sup>10</sup>

### 2.7.2 Analysis

All samples must be analyzed initially for asbestos content by PLM. XRD should be used as an auxiliary method when a second, independent analysis is requested.

**Note:** Asbestos is a toxic substance. All handling of dry materials should be performed in an operating fume hood.

#### 2.7.2.1 Sample Preparation

The method of sample preparation required for XRD analysis will depend on: (1) the condition of the sample received (sample size, homogeneity, particle size distribution, and overall composition as determined by PLM), and (2) the type of XRD analysis to be performed (qualitative or quantitative; thin layer or bulk).

Bulk materials are usually received as inhomogeneous mixtures of complex composition with very wide particle size distributions. Preparation of a homogeneous, representative sample from asbestos-containing materials is particularly difficult because the fibrous nature of the asbestos minerals inhibits mechanical mixing and stirring, and because milling procedures may cause adverse lattice alterations.

A discussion of specific matrix reduction procedures is given below. Complete methods of sample preparation are detailed in Sections 2.7.2.2 and 2.7.2.3. **Note:** All samples should be examined microscopically before and after each matrix reduction step to monitor changes in sample particle size distribution, composition, and crystallinity, and to ensure sample representativeness and homogeneity for analysis.

**2.7.2.1.1 Milling—Mechanical** milling of asbestos materials has been shown to decrease fiber crystallinity, with a resultant decrease in diffraction intensity of the specimen; the degree of lattice alteration is related to the duration and type of milling process.<sup>19-22</sup> Therefore, all milling times should be kept to a minimum.

For qualitative analysis, particle size is not usually of critical importance and initial characterization of the material with a minimum of matrix reduction is often desirable to document the composition of the sample as received. Bulk samples of very large particle size (>2-3 mm) should be comminuted to ~100  $\mu$ m. A mortar and pestle can sometimes be used in size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced by cutting with a razor blade in a mortar, or by grinding in a suitable mill (e.g., a microhammer mill or equivalent). When using a mortar for grinding or cutting, the sample should be moistened with ethanol, or some other suitable wetting agent, to minimize exposures.

For accurate, reproducible quantitative analysis, the particle size of both sample and standard materials should be reduced to ~10  $\mu$ m (see Section 2.3.3). Dry ball milling at liquid nitrogen temperatures (e.g., Spex Freezer Mill, or equivalent) for a maximum time of 10 min is recommended to obtain satisfactory particle size distributions while protecting the integrity of the crystal lattice.<sup>6</sup> Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to <10  $\mu$ m.<sup>6,10</sup>

Final particle size distributions should always be verified by optical microscopy or another suitable method.

#### 2.7.2.1.2. Low temperature ashing—

For materials shown by PLM to contain large amounts of gypsum, cellulose, or other organic materials, it may be desirable to ash the samples prior to analysis to reduce background radiation or matrix interference. Since chrysotile undergoes dehydroxylation at temperatures between 550°C and 650°C, with subsequent transformation to forsterite,<sup>23,24</sup> ashing temperatures should be kept below 500°C. Use of a low temperature asher is recommended. In all cases, calibration of the oven is essential to ensure that a maximum ashing temperature of 500°C is not exceeded.

**2.7.2.1.3 Acid leaching—**Because of the interference caused by gypsum and some carbonates in the detection of asbestiform minerals by XRD (see Section 2.3.1), it may be necessary to remove these interferents by a simple acid leaching procedure prior to analysis (see Section 1.7.2.2).

### 2.7.2.2 Qualitative Analysis

**2.7.2.2.1 Initial screening of bulk material—**Qualitative analysis should be performed on a representative, homogeneous portion of the sample with a minimum of sample treatment using the following procedure:

1. Grind and mix the sample with a mortar and pestle (or equivalent method, see Section 2.7.2.1.1) to a final particle size sufficiently small (~100  $\mu$ m) to allow adequate packing into the sample holder.
2. Pack sample into a standard bulk sample holder. Care should be taken to ensure that a representative portion of the milled sample is selected for analysis. Particular care should be taken to avoid possible size segregation of the sample. (Note: Use of a back-packing method<sup>25</sup> for bulk sample



preparation may reduce preferred orientation effects.)

3. Mount the sample on the diffractometer and scan over the diagnostic peak regions for the serpentine (~7.4 Å) and amphibole (8.2-8.5 Å) minerals (see Table 2-2). The X-ray diffraction equipment should be optimized for intensity. A slow scanning speed of 1° 2θ/min is recommended for adequate resolution. Use of a sample spinner is recommended.
4. Submit all samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals to a full qualitative XRD scan (5°-60° 2θ, 1° 2θ/min) to verify initial peak assignments and to identify potential matrix interferences when subsequent quantitative analysis is to be performed.
5. Compare the sample XRD pattern with standard reference powder diffraction patterns (i.e., JCPDS powder diffraction data<sup>3</sup> or those of other well-characterized reference materials). Principal lattice spacings of asbestiform minerals are given in Table 2-2; common constituents of bulk insulation and wall materials are listed in Table 2-3.

**2.7.2.2.2 Detection of minor or trace constituents**—Routine screening of bulk materials by XRD may fail to detect small concentrations (<5 percent) of asbestos. The limits of detection will, in general, be improved if matrix absorption effects are minimized, and if the sample particle size is reduced to the optimal 1 to 10 μm range, provided that the crystal lattice is not degraded in the milling process. Therefore, in those instances where confirmation of the presence of an asbestiform mineral at very low levels is required, or where a negative result from initial screening of the bulk material by XRD (see Section 2.7.2.2.1) is in conflict with previous PLM results, it may be desirable to prepare the sample as described for quantitative analysis (see Section 2.7.2.3) and step-scan over appropriate 2θ ranges of selected diagnostic peaks (Table 2-2). Accurate transfer of the sample to the silver membrane filter is not necessary unless subsequent quantitative analysis is to be performed.

#### 2.7.2.3 Quantitative Analysis

The proposed method for quantitation of asbestos in bulk samples is a modification of the NIOSH-recommended thin-layer method for chrysotile in air.<sup>5</sup> A thick-layer or bulk

method involving pelletizing the sample may be used for semiquantitative analysis;<sup>7,8</sup> however, this method requires the addition of an internal standard, use of a specially fabricated sample press, and relatively large amounts of standard reference materials. Additional research is required to evaluate the comparability of thin- and thick-layer methods for quantitative asbestos analysis.

For quantitative analysis by thin-layer methods, the following procedure is recommended.

1. Mill and size all or a substantial representative portion of the sample as outlined in Section 2.7.2.1.1.
2. Dry at 100°C for 2 hr; cool in a desiccator.
3. Weigh accurately to the nearest 0.01 mg.
4. Samples shown by PLM to contain large amounts of cellulosic or other organic materials, gypsum, or carbonates, should be submitted to appropriate matrix reduction procedures described in Sections 2.7.2.1.2 and 2.7.2.1.3. After ashing and/or acid treatment, repeat the drying and weighing procedures described above, and determine the percent weight loss,  $L$ .
5. Quantitatively transfer an accurately weighed amount (50-100 mg) of the sample to a 1-L volumetric flask with approximately 200 mL isopropanol to which 3 to 4 drops of surfactant have been added.
6. Ultrasonicate for 10 min at a power density of approximately 0.1 W/mL, to disperse the sample material.
7. Dilute to volume with isopropanol.
8. Place flask on a magnetic stirring plate. Stir.
9. Place a silver membrane filter on the filtration apparatus, apply a vacuum, and attach the reservoir. Release the vacuum and add several milliliters of isopropanol to the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension so that total sample weight,  $W_t$ , on the filter will be approximately 1 mg. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a

clean pipet. Transfer the aliquot to the reservoir. Filter rapidly under vacuum. Do not wash the reservoir walls. Leave the filter apparatus under vacuum until dry. Remove the reservoir, release the vacuum, and remove the filter with forceps. (Note: Water-soluble matrix interferences such as gypsum may be removed at this time by careful washing of the filtrate with distilled water. Extreme care should be taken not to disturb the sample.)

10. Attach the filter to a flat holder with a suitable adhesive and place on the diffractometer. Use of a sample spinner is recommended.
11. For each asbestos mineral to be quantitated, select a reflection (or reflections) that has been shown to be free from interferences by prior PLM or qualitative XRD analysis and that can be used unambiguously as an index of the amount of material present in the sample (see Table 2-2).
12. Analyze the selected diagnostic reflection(s) by step scanning in increments of 0.02° 2θ for an appropriate fixed time and integrating the counts. (A fixed count scan may be used alternatively; however, the method chosen should be used consistently for all samples and standards.) An appropriate scanning interval should be selected for each peak, and background corrections made. For a fixed time scan, measure the background on each side of the peak for one-half the peak-scanning time. The net intensity,  $I_n$ , is the difference between the peak integrated count and the total background count.
13. Determine the net count,  $I_{n0}$ , of the filter 2.36 Å silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak,  $I_{n0}^*$ . Scan times may be less for measurement of silver peaks than for sample peaks; however, they should be constant throughout the analysis.
14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 Å peak of an α-quartz reference crystal). After each unknown is scanned, determine the net



count,  $I_P$ , of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by  $I_P$ .

$$I_s = \frac{I_s}{I_P}, I_{As} = \frac{I_{As}}{I_P}, \text{ and } I_{Ls} = \frac{I_{Ls}}{I_P}$$

## 2.8 Calibration

### 2.8.1 Preparation of Calibration Standards

1. Mill and size standard asbestos materials according to the procedure outlined in Section 2.7.2.1.1. *Equivalent, standardized matrix reduction and sizing techniques should be used for both standard and sample materials.*
2. Dry at 100°C for 2 hr; cool in a desiccator.
3. Prepare two suspensions of each standard in isopropanol by weighing approximately 10 and 50 mg of the dry material to the nearest 0.01 mg. Quantitatively transfer each to a 1-L volumetric flask with approximately 200 mL isopropanol to which a few drops of surfactant have been added.
4. Ultrasonicate for 10 min at a power density of approximately 0.1 W/mL to disperse the asbestos material.
5. Dilute to volume with isopropanol.
6. Place the flask on a magnetic stirring plate. Stir.
7. Prepare, in triplicate, a series of at least five standard filters to cover the desired analytical range, using appropriate aliquots of the 10 and 50 mg/L suspensions. For each standard, mount a silver membrane filter on the filtration apparatus. Place a few mL of isopropanol in the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Keep the tip of the pipet near the surface of the isopropanol. Filter rapidly under vacuum. Do not wash the sides of the reservoir. Leave the vacuum on for a time sufficient to dry the filter. Release the vacuum and remove the filter with forceps.

### 2.8.2 Analysis of Calibration Standards

1. Mount each filter on a flat holder. Perform step scans on selected diagnostic reflections of the standards and reference specimen using the procedure outlined in Section 2.7.2.3, step 12, and the same conditions as those used for the samples.
2. Determine the normalized intensity for each peak measured,  $I_{sm}$ , as outlined in Section 2.7.2.3, step 14.

## 2.9 Calculations

For each asbestos reference material, calculate the exact weight deposited on each standard filter from the concentrations of the standard suspensions and aliquot volumes. Record the weight,  $w$ , of each standard. Prepare a calibration curve by regressing  $I_{sm}$  on  $w$ . Poor reproducibility ( $\pm 15$  percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight line equation.

Determine the slope,  $m$ , of the calibration curve in counts/microgram. The intercept,  $b$ , of the line with the  $I_{sm}$  axis should be approximately zero. A large negative intercept indicates an error in determining the background. This may arise from incorrectly measuring the baseline or from interference by another phase at the angle of background measurement. A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

Using the normalized intensity,  $I_{As}$ , for the attenuated silver peak of a sample, and the corresponding normalized intensity from the unattenuated silver peak,  $I_{Ls}$ , of the sample filter, calculate the transmittance,  $T$ , for each sample as follows:

$$T = \frac{I_{Ls}}{I_{As}}$$

Determine the correction factor,  $f(T)$ , for each sample according to the formula:

$$f(T) = \frac{-R(\ln T)}{1-T^R}$$

where

$$R = \frac{\sin \theta_{As}}{\sin \theta_s}$$

$\theta_{As}$  = angular position of the measured silver peak (from Bragg's Law), and  
 $\theta_s$  = angular position of the diagnostic asbestos peak.

Calculate the weight,  $W_s$ , in micrograms, of the asbestos material analyzed for in each sample, using the appropriate calibration data and absorption corrections:

$$W_s = \frac{I_s f(T) - b}{m}$$

Calculate the percent composition,  $P_s$ , of each asbestos mineral analyzed for in the parent material, from the total sample weight,  $W_T$ , on the filter:

$$P_s = \frac{W_s (1 - 0.01L)}{W_T} \times 100$$

where

$P_s$  = percent asbestos mineral in parent material,  
 $W_s$  = mass of asbestos mineral on filter, in  $\mu\text{g}$ ;  
 $W_T$  = total sample weight on filter, in  $\mu\text{g}$ ;  
 $L$  = percent weight loss of parent material on ashing and/or acid treatment (see Section 2.7.2.3).

## 2.10 References

1. H. P. Klug and L. E. Alexander, *X-ray Diffraction Procedures for Polycrystalline and Amorphous Materials*, 2nd ed., New York: John Wiley and Sons, 1979.
2. L. V. Azaroff and M. J. Buerger, *The Powder Method of X-ray Crystallography*, New York: McGraw-Hill, 1958.
3. JCPDS-International Center for Diffraction Data Powder Diffraction File, on Powder Diffraction Studies, 1601 Park Lane, Swarthmore, PA.
4. W. J. Campbell, C. W. Huggins, and A. G. Wylie, *Chemical and Physical Characterization of Amosite, Chrysotile, Crocidolite, and Nonfibrous Tremolite for National Institute of Environmental Health Sciences Oral Ingestion Studies*, U.S. Bureau of Mines Report of Investigation RI8452, 1980.
5. B. A. Lange and J. C. Haartz, Determination of microgram quantities of asbestos by X-ray diffraction: Chrysotile in thin dust layers of matrix material, *Anal. Chem.*, 51(4):520-525, 1979.
6. NIOSH Manual of Analytical Methods, Volume 5, U.S. Dept. HEW, August 1979, pp. 309-1 to 309-9.
7. H. W. Dunn and J. H. Stewart, Jr., Determination of chrysotile in building materials by X-ray Diffractometry, *Anal. Chem.*, 54(7): 1122-1125, 1982.



8. M. Taylor, Methods for the quantitative determination of asbestos and quartz in bulk samples using X-ray diffraction, *The Analyst*, 103(1231):1009-1020, 1978.
9. L. Birks, M. Fatemi, J. V. Gilfrich, and E. T. Johnson, *Quantitative Analysis of Airborne Asbestos by X-ray Diffraction*, Naval Research Laboratory Report 7879, Naval Research Laboratory, Washington, DC, 1975.
10. U.S. Environmental Protection Agency, *Asbestos-Containing Materials in School Buildings: A Guidance Document*, Parts 1 and 2, EPA/OTS No. C00090, March 1979.
11. J. B. Krause and W. H. Ashton, Misidentification of asbestos in talc, pp. 339-353, In: *Proceedings of Workshop on Asbestos: Definitions and Measurement Methods* (NBS Special Publication 506), C. C. Gravatt, P. D. LaFleur, and K. F. Heinrich (eds.), Washington, DC: National Measurement Laboratory, National Bureau of Standards, 1977 (issued 1978).
12. H. D. Stanley, The detection and identification of asbestos and asbestiform minerals in talc, pp. 325-337, In: *Proceedings of Workshop on Asbestos: Definitions and Measurement Methods* (NBS Special Publication 506), C. C. Gravatt, P. D. LaFleur, and K. F. Heinrich (eds.), Washington, DC: National Measurement Laboratory, National Bureau of Standards, 1977 (issued 1978).
13. A. L. Rickards, Estimation of trace amounts of chrysotile asbestos by X-ray diffraction, *Anal. Chem.*, 44(11):1872-3, 1972.
14. P. M. Cook, P. L. Smith, and D. G. Wilson, Amphibole fiber concentration and determination for a series of community air samples: Use of X-ray diffraction to supplement electron microscope analysis, In: *Electron Microscopy and X-ray Applications to Environmental and Occupation Health Analysis*, P. A. Russell and A. E. Hutchings (eds.), Ann Arbor: Ann Arbor Science Publications, 1977.
15. A. N. Rohl and A. M. Langer, Identification and quantitation of asbestos in talc, *Environ. Health Perspectives*, 9:95-109, 1974.
16. J. L. Graf, P. K. Ase, and R. G. Draftz, *Preparation and Characterization of Analytical Reference Minerals*, DHEW (NIOSH) Publication No. 79-139, June 1979.
17. J. C. Haartz, B. A. Lange, R. G. Draftz, and R. F. Scholl, Selection and characterization of fibrous and nonfibrous amphiboles for analytical methods development, pp. 295-312, In: *Proceedings of Workshop on Asbestos: Definitions and Measurement Methods* (NBS Special Publication 506), C. C. Gravatt, P. D. LaFleur, and K. F. Heinrich (eds.), Washington, DC: National Measurement Laboratory, National Bureau of Standards, 1977 (issued 1978).
18. Personal communication, A. M. Langer, Environmental Sciences Laboratory, Mount Sinai School of Medicine of the City University of New York, New York, NY.
19. A. M. Langer, M. S. Wolff, A. N. Rohl, and I. J. Selikoff, Variation of properties of chrysotile asbestos subjected to milling, *J. Toxicol. and Environ. Health*, 4:173-188, 1978.
20. A. M. Langer, A. D. Mackler, and F. D. Pooley, Electron microscopical investigation of asbestos fibers, *Environ. Health Perspect.*, 9:63-80, 1974.
21. E. Occella and G. Maddalon, X-ray diffraction characteristics of some types of asbestos in relation to different techniques of comminution, *Med. Lavoro*, 54(10):628-636, 1963.
22. K. R. Spurny, W. Stober, H. Opiela, and G. Weiss, On the problem of milling and ultrasonic treatment of asbestos and glass fibers in biological and analytical applications, *Am. Ind. Hyg. Assoc. J.*, 41:198-203, 1980.
23. L. G. Berry and B. Mason, *Mineralogy*, San Francisco: W. H. Freeman & Co., 1959.
24. J. P. Schelz, The detection of chrysotile asbestos at low levels in talc by differential thermal analysis, *Thermochimica Acta*, 8:197-204, 1974.
25. Reference 1, pp. 372-374.
26. J. Leroux, *Staub-Reinheit Luft*, 29.26 (English), 1969.
27. J. A. Leroux, B. C. Davey, and A. Paillard, *Am. Ind. Hyg. Assoc. J.*, 34:409, 1973.

**Table 2-1. The asbestos minerals and their nonasbestiform analogs.**

Asbestiform	Nonasbestiform
<b>Serpentine</b>	
Chrysotile	Antigorite, lizardite
<b>Amphibole</b>	
Anthophyllite asbestos	Anthophyllite
Cummingtonite-grunerite asbestos ("Amosite")	Cummingtonite-grunerite
Crocidolite	Riebeckite
Tremolite asbestos	Tremolite
Actinolite asbestos	Actinolite

**Table 2-2. Principal lattice spacings of asbestiform minerals<sup>a</sup>**

Minerals	JCPDS Principal d-spacings (Å) and relative intensities				Powder diffraction file <sup>c</sup> number
Chrysotile	7.37 <sub>100</sub>	3.65 <sub>70</sub>	4.57 <sub>80</sub>	21-543 <sup>c</sup>	25-645
	7.36 <sub>100</sub>	3.66 <sub>80</sub>	2.45 <sub>85</sub>		22-1162 (theoretical)
	7.10 <sub>100</sub>	2.33 <sub>80</sub>	3.55 <sub>70</sub>		17-745 (nonfibrous)
"Amosite"	8.33 <sub>100</sub>	3.06 <sub>70</sub>	2.756 <sub>70</sub>		27-1170 (UICC)
	8.22 <sub>100</sub>	3.060 <sub>85</sub>	3.25 <sub>70</sub>		
Anthophyllite	3.05 <sub>100</sub>	3.24 <sub>80</sub>	8.26 <sub>55</sub>	9-455	
	3.06 <sub>100</sub>	8.33 <sub>70</sub>	3.23 <sub>80</sub>	16-401 (synthetic)	
Actinolite	2.72 <sub>100</sub>	2.54 <sub>100</sub>	3.40 <sub>80</sub>	25-157	
Crocidolite	8.35 <sub>100</sub>	3.10 <sub>85</sub>	2.720 <sub>55</sub>	27-1415 (UICC)	
Tremolite	8.38 <sub>100</sub>	3.12 <sub>100</sub>	2.705 <sub>80</sub>	13-437 <sup>c</sup>	
	2.706 <sub>100</sub>	3.14 <sub>85</sub>	8.43 <sub>80</sub>	20-1310 <sup>c</sup> (synthetic)	
	3.13 <sub>100</sub>	2.706 <sub>80</sub>	8.44 <sub>80</sub>	23-666 (synthetic mixture with richterite)	

<sup>a</sup>This information is intended as a guide, only. Complete powder diffraction data, including mineral type and source, should be referred to, to ensure comparability of sample and reference materials where possible. Additional precision XRD data on amosite, crocidolite, tremolite, and chrysotile are available from the U.S. Bureau of Mines, Reference 4.

<sup>b</sup>From Reference 3.

<sup>c</sup>Fibrosity questionable.



**Table 2-3. Common constituents in insulation and wall materials (from Ref. 10)**

A. Insulation materials	B. Spray finishes or paints
Chrysotile	Bassanite
"Amosite"	Carbonate minerals (calcite, dolomite, vaterite)
Crocidolite	Talc
*Rock wool	Tremolite
*Slag wool	Anthophyllite
*Fiber glass	Serpentine (including chrysotile)
Gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ )	*Amosite"
Vermiculite (micas)	Crocidolite
*Perlite	*Mineral wool
Clays (kaolin)	*Rock wool
*Wood pulp	*Slag wool
*Paper fibers (talc, clay, carbonate fillers)	*Fiber glass
Calcium silicates (synthetic)	Clays (kaolin)
Opagues (chromite, magnetite inclusions in serpentine)	Micas
Hematite (inclusions in "amosite")	Chlorite
Magnesite	Gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ )
*Diatomaceous earth	Quartz
	*Organic binders and thickeners
	Hydromagnesite
	Wollastonite
	Opagues (chromite, magnetite inclusions in serpentine)
	Hematite (inclusions in "amosite")

\*Amorphous materials--contribute only to overall scattered radiation and increased background radiation.

**Table 2-4. Interferences in XRD analysis of asbestiform minerals**

Asbestiform mineral	Primary diagnostic peaks (approximate d-spacings in Å)	Interference
Serpentine		
Chrysotile	7.4	Nonasbestiform serpentines (antigorite, lizardite)
		Chlorite
		Kaolinite
		Gypsum
	3.7	Nonasbestiform serpentines (antigorite, lizardite)
		Chlorite
		Halloysite
		Cellulose
Amphibole		
"Amosite"	3.1	Nonasbestiform amphiboles (cummingtonite, grunerite, anthophyllite, riebeckite, tremolite)
Anthophyllite		Mutual interferences
Crocidolite		Carbonates
Tremolite		Talc
	8.3	Nonasbestiform amphiboles (cummingtonite, grunerite, anthophyllite, riebeckite, tremolite)
		Mutual interferences

United States  
Environmental Protection  
Agency

Center for Environmental Research  
Information  
Cincinnati OH 45268

Official Business  
Penalty for Private Use \$300

**APPENDIX III**  
**Preliminary Assessment Report**

Suite 420, 120 Howard Street, San Francisco, CA 94105 • (415) 513-5022

TECHNICAL ASSISTANCE TEAM FOR EMERGENCY RESPONSE REMOVAL AND PREVENTION  
FBI CONTACT: 62-01-622

December 3, 1965

U.S. Environmental Protection Agency  
215 Fremont Street  
San Francisco, CA 94105

PCS# 5033  
TDD# 098511-11  
TAT# 098512-F-0002

Attention: P.T. Brubaker

Subject: Copperopolis

Gentlemen:

About 20 years ago fill dirt from the Calaveras Asbestos Mine was imported to the town of Copperopolis, CA to provide road bed material for a newly constructed development. No "clean" soil was mixed with the asbestos tailings, probably since asbestos-related health problems are a relatively recent discovery. The owner of the mine 20 years ago, Mr. H.K. Porter (he is not the present owner), now reportedly works in Pittsburgh, PA for the H.K. Porter Company.

Ron Shields, a representative of the Copopolis Community Board, alerted the EPA's Emergency Response Section (ERS) to the asbestos problem. As a result of this, Technical Assistance Team (TAT) member Steve Wolfe visited the site and was informed of the problem, in detail, by Ron Shields. Upon arrival at the site, it was determined that approximately 15 miles of dirt roads exist within the community. Adequate soil sample coverage from only a one-day visit was thus obviously a problem. In order to circumvent this dilemma, a plan was designed that would include sampling the most heavily travelled roads (which therefore generate the most airborne asbestos fibers) coupled with the areas containing the heaviest population. For the most part, intersections of the dirt roads best fit these criteria.

A total of 29 surface soil samples were gathered. The locations are designated on a map which has been forwarded to Dan Shane, (On-Scene-Coordinator). All of the samples were taken from road surfaces except numbers one and 13. Number one was taken from a drain pipe. The water from this pipe drains into a small stream that originates in the area. This stream is a source of drinking water for the community. The water is gathered from a spring, and the water of the spring is used for drinking. The soil from the drain pipe is used for growing plants. This plant is used for a variety of purposes for about the past 10 years.

000123





Page 2  
P.T. Brubaker  
T.T #093512-F-0002

The laboratory results for the samples (performed by EAL Corporation) are as follows:

Sample 1-29: 10-20% chrysotile asbestos. The matrix material consists primarily of clay minerals, quartz, and some calcite.

The accompanying photographs present an overall view of the community as well as a closer look at some of the sampled areas.

If you have any further questions regarding this matter please do not hesitate to contact me.

Respectfully submitted,

Steven P. Wolfe  
Technical Assistance Team Member

cc: NED  
File

**APPENDIX IV**  
**Laboratory Analysis Reports**



**Thermo Electron  
EAL Corporation**

2030 Wright Avenue  
Richmond California 94804  
(415) 235-2633  
(TWX) 910-382-8132

**ANALYSIS REPORT**

RIEDEL ENVIRONMENTAL  
SERVICES  
230 CUTTING BLVD.  
RICHMOND, CA 94802

DATE: 5-12-86  
Samples Received: 5-2-86  
EAL W.O. No. 17994-0  
Project Name: Copperopolis, CA

SAMPLE IDENTIFICATION		ASBESTOS		OTHER COMPONENTS	
EAL	CUSTOMER	%	TYPE	%	TYPE
2720-95-1	S001	30-40	chrysotile	60-70	antigorite clay particles misc. particles
2720-95-2	S002	<1	chrysotile	99+	quartz feldspar clay particles fe-oxide misc. particles
2720-95-3	S003	<1	none detected	100	quartz feldspar clay particles fe-oxide misc. particles
2720-95-4	S004	<1	chrysotile	99+	quartz feldspar clay particles fe-oxide misc. particles
2720-95-5	S005	30-40	chrysotile	60-70	antigorite clay particles feldspar misc. particles
2720-95-6	S006	5-10	chrysotile	90-95	quartz feldspar fe-oxide clay particles antigorite misc. particles
2720-95-7	S007	40-50	chrysotile	50-60	antigorite clay particles misc. particles

000126






2030 Wright Avenue  
Richmond, California 94804  
(415) 235-2633  
(TAX) 910-382-8132

Report to: RIEDEL ENVIRONMENTAL

SAMPLE IDENTIFICATION		ASBESTOS		OTHER COMPONENTS	
EAL	CUSTOMER	%	TYPE	%	TYPE
2720-95-8	S008	30-40	chrysotile	60-70	quartz feldspar antigorite misc. particles
2720-95-9	S009	30-40	chrysotile	60-70	quartz feldspar antigorite misc. particles
2720-95-10	S0010	40-50	chrysotile	50-60	quartz feldspar antigorite misc. particles
2720-95-11	S0011	20-30	chrysotile	70-80	quartz feldspar antigorite misc. particles
2720-95-12	S0012	<1	none detected	100	quartz feldspar clay particles misc. particles

Analysis by polarized light microscopy

  
Jesse E. Fisher  
Program Manager

EAL Corporation laboratories are Accredited by the American Industrial Hygiene Association; approved by the State of California for complete chemical, radiological, bacteriological, and bioassay analyses.

000127



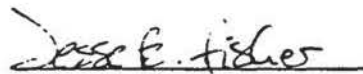
# Thermo Electron EAL Corporation

2030 Wright Avenue  
Richmond, California 94804  
(415) 235-2533  
(TX) 910-352-8132

Report to: RIEDEL ENVIRONMENTAL

SAMPLE IDENTIFICATION		ASBESTOS		OTHER COMPONENTS	
EAL	CUSTOMER	%	TYPE	%	TYPE
2720-95-8	S008	30-40	chrysotile	60-70	quartz feldspar antigorite misc. particles
2720-95-9	S009	30-40	chrysotile	60-70	quartz feldspar antigorite misc. particles
2720-95-10	S0010	40-50	chrysotile	50-60	quartz feldspar antigorite misc. particles
2720-95-11	S0011	20-30	chrysotile	70-80	quartz feldspar antigorite misc. particles
2720-95-12	S0012	<1	none detected	100	quartz feldspar clay particles misc. particles
2720-96-11	S015	40-50	chrysotile	50-60	antigorite clay particles feldspar misc. particles

Analysis by polarized light microscopy

  
Jesse E. Fisher  
Program Manager

EAL Corporation laboratories are Accredited by the American Industrial Hygiene Association; approved by the State of California for complete chemical, radiological, bacteriological, and bioassay analyses.

000128



2030 Wright Avenue  
Richmond, California 94804  
(415) 235-2633  
(TWX) 910-382-8132

# ANALYSIS REPORT

RECEIVED

MAY 20 1986

TAT REG IX

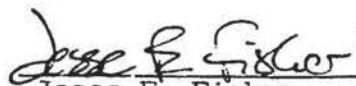
RIEDEL ENVIRONMENTAL  
SERVICES  
230 CUTTING BLVD.  
RICHMOND, CA 94802

DATE: 5-22-86  
Samples Received: 5-16-86  
EAL W.O. No. 17994-0  
Job No. COPPESOPOLIS

SAMPLE IDENTIFICATION		ASBESTOS		OTHER COMPONENTS	
EAL	CUSTOMER	%	TYPE	%	TYPE
2720-99-1	S019	10-20	chrysotile	80-90	antigorite lizardite clay particles feldspar quartz misc. particles
2720-99-2	S020	5-10	chrysotile	90-95	antigorite lizardite clay particles feldspar quartz iron oxides misc. particles
2720-99-3	S021	3-5	chrysotile	95-97	antigorite lizardite clay particles feldspar quartz misc. particles

Analysis by polarized light microscopy

Results were reported to Dan Shane on 5/21/86

  
Jesse E. Fisher  
Program Manager

000129

EAL Corporation laboratories are Accredited by the American Industrial Hygiene Association; approved by the State of California for complete chemical, radiological, bacteriological, and biological analysis.





2030 Wright Avenue  
Richmond, California 94804  
(415) 235-2633  
(TWX) 910-382-3132

# ANALYSIS REPORT


RECEIVED  
JUN 9 1986  
TAT REG IX

RIEDEL ENVIRONMENTAL  
SERVICES  
230 CUTTING BLVD.  
RICHMOND, CA 94802

DATE: 6-3-86  
Samples Received: 6-2-86  
EAL W.O. No. 17994-0  
Project: COPPEROPOLIS

SAMPLE IDENTIFICATION		ASBESTOS		OTHER COMPONENTS	
EAL	CUSTOMER	%	TYPE	%	TYPE
=====					
2720-101-1	95022232 S022	10-20	chrysotile	80-90	magnetite antigorite misc. particles

Analysis by polarized light microscopy

  
Jesse E. Fisher  
Program Manager

EAL Corporation laboratories are Accredited by the American Industrial Hygiene Association; approved by the State of California for complete chemical, radiological, bacteriological, and bioassay analyses.

000130

# **Thermo Electron EAL Corporation**

2030 Wright Avenue  
Richmond California 94804  
(415) 235-2633  
(TWX) 910-382-8132

## ANALYSIS REPORT

RIEDEL ENVIRONMENTAL  
SERVICES  
230 CUTTING BLVD.  
RICHMOND, CA 94802  
ATTN: GARY FLOYD

DATE: 5-14-86  
Samples Received: 5-8-86  
EAL W.O. No. 17994-0  
Purchase Order No.

SAMPLE IDENTIFICATION		ASBESTOS FIBER COUNT		VOLUME
EAL	CUSTOMER	fibers/filter	fibers/cc	(liter)
2720-96-1	A001-CONTROL SE SIDE	<1100.		
2720-96-2	A002-CONTROL NW SIDE	<1100.		
2720-96-3	A003	<1100.	<0.00036	3000.
2720-96-4	A004	<1100.	<0.00036	3000.
2720-96-5	A005	<1100.	<0.00052	2070.
2720-96-6	A006	<1100.	<0.00036	3000.
2720-96-7	A007	<1100.	<0.00036	3000.
2720-96-8	A008	<1100.	<0.00036	3000.
2720-96-9	A009	<1100.	<0.00036	3000.
2720-96-10	A010	<1100.	<0.00036	3000.

Analysis by phase contrast microscopy per NIOSH P&CAM 239

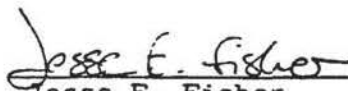
SAMPLE IDENTIFICATION		ASBESTOS		OTHER COMPONENTS	
EAL	CUSTOMER	%	TYPE	%	TYPE
2720-96-11	S015	40-50	chrysotile	50-60	antigorite clay particles feldspar misc. particles

Analysis by polarized light microscopy

**RECEIVED**

41 JUN 1986

**TAT REG IX**

  
Jesse E. Fisher  
Program Manager

EAL Corporation laboratories are Accredited by the American Industrial Hygiene Association; approved by the State of California for complete chemical, radiological, bacteriological, and bioassay analyses. 000131



**Thermo Electron  
EAL Corporation**

2030 Wright Avenue  
Richmond, California 94804  
(415) 235-2633  
(TWX) 910-382-8132

**ANALYSIS REPORT**

**RIEDEL ENVIRONMENTAL  
SERVICES**  
230 CUTTING BLVD.  
RICHMOND, CA 94802

DATE: 5-16-86  
Samples Received: 5-12-86  
EAL W.O. No. 17994-0  
Project: COPPEROPOLIS

SAMPLE IDENTIFICATION		ASBESTOS FIBER COUNT		VOLUME
EAL	CUSTOMER	fibers/filter	fibers/cc	(liter)
2720-97-1	A011	<1000.	<0.00034	3000.
2720-97-2	A012	<1000.	<0.00034	3000.
2720-97-3	A013	<1000.	<0.00034	3000.
2720-97-4	A014	<1000.	<0.00034	3000.
2720-97-5	A015	<1000.	<0.00034	3000.
2720-97-6	TA002	18000.	0.0090	2004.
2720-97-7	TA003	2000.	0.0010	2004.
2720-97-8	TA004	35000.	0.017	2004.
2720-97-9	TA005	<1000.	<0.00050	2004.
2720-97-10	A016	4500.	0.0015	3000.
2720-97-11	A017	<1000.	<0.00034	3000.
2720-97-12	A018	2000.	0.00067	3000.
2720-97-13	A019	1000.	0.00033	3000.
2720-97-14	A020	1000.	0.00033	3000.
2720-97-15	A021	1000.	0.00033	3000.
2720-97-16	A022	1000.	0.00033	3000.

Analysis by phase contrast microscopy per NIOSH P&CAM 239

  
Jesse E. Fisher  
Program Manager

EAL Corporation laboratories are Accredited by the American Industrial Hygiene Association; approved by the State of California for complete chemical, radiological, bacteriological, and bioassay analyses. 000132



Med-Tox Associates, Inc. 1229 Morena Blvd San Diego, CA 92110 (619) 276-8843

TEM QUANTITATIVE ANALYSIS  
(AIR)

ROY F. WESTON, INC.  
120 HOWARD ST., S.420 SAN FRANCISCO, CA 415-543-5099  
GARY A. FLOYD

Job Number : 5112  
Sample Number : TA006  
MT Number : 10663  
Date : JUNE 23, 1986  
Description : COPPEROPOLIS, CA

Total Area Scanned : 23940.0 sq um  
Magnification : 20000 x  
Area of Original Filter : 8.55 sq cm  
Area Ashed : 4.28 sq cm  
Dilution Factor : 1.00  
Area of Membrane : 2.01 sq cm  
Volume of Air : 2.004 cubic meter  
Total number of structs : 118  
Detection Limit : 8369.4 fibers/cu meter (1 fiber)  
Reliable Detection Limit: 41847.2 fibers/cu meter (5 fibers)

Asbestos Summary

	Fiber	Bundle	Cluster	Matrix
Structure Conc. (structures/cu meter)				
Chrysotile	251083.3	426841.6	175758.3	0.0
Amphibole	0.0	0.0	0.0	0.0
Mass Concentration (ng/cu meter)				
Chrysotile	20.16	210.95	119815.53	0.00
Amphibole	0.00	0.00	0.00	0.00

TOTAL ASBESTOS STRUCTURES:

Structure Concentration 853683.1 (structures/cu meter)  
Mass Concentration 120046.63 (ng/cu meter)

Analyst Tom Sagerhart Date 6, 23, 86

000133

Med-Tox Associates, Inc. 1229 Morena Blvd San Diego, CA 92110 (619) 276-8843

TEM QUANTITATIVE ANALYSIS  
(AIR)

ROY F. WESTON, INC.  
120 HOWARD ST., S.420 SAN FRANCISCO, CA 415-543-5099  
GARY A. FLOYD

Job Number : 5112  
Sample Number : TA006  
MT Number : 10663  
Date : JUNE 23, 1986  
Description : COPPERPOLIS, CA

Total Area Scanned : 23940.0 sq um  
Magnification : 20000 x  
Area of Original Filter : 8.55 sq cm  
Area Ashed : 4.28 sq cm  
Dilution Factor : 1.00  
Area of Membrane : 2.01 sq cm  
Volume of Air : 2.004 cubic meter  
Total number of structs : 118  
Detection Limit : 8369.4 fibers/cu meter (1 fiber)  
Reliable Detection Limit: 41847.2 fibers/cu meter (5 fibers)

\* \* FIBER \* \*

	Chrysotile	Amphibole	Indeterminant	Nonasbestos
Total Number of Structures Counted	30	0	1	5
Percent Total Structures	23.4	0.0	0.8	4.2
Total Structure Concentration (structures per cubic meter of air)	251083.3	0.0	8369.4	41847.2
Total Surface Area (sq um per cubic meter of air)	134917.9	0.0	3976.9	13376.8
Total Mass Concentration (ng per cubic meter of air)	20.16	0.00		
Structures > 5.0 um length Counted	1	0	0	0
Percent Structures > 5.0 um length	0.8	0.0	0.0	0.0
Struct Concentration > 5.0 um length (structures per cubic meter of air)	8369.4	0.0	0.0	0.0
Length geo. mean	1.3645	0.0000	3.0000	1.1933
standard deviation	1.3405	0.0000	0.0000	0.6262
Diameter geo. mean	0.0585	0.0000	0.0500	0.0758
standard deviation	0.0835	0.0000	0.0000	0.0671

00C134

Med-Tox Associates, Inc. 1229 Morena Blvd San Diego, CA 92110 (619) 276-8843

TEM QUANTITATIVE ANALYSIS  
(AIR)

ROY F. WESTON, INC.  
120 HOWARD ST., S. 420 SAN FRANCISCO, CA 415-543-5099  
GARY A. FLOYD

Job Number : 5112  
Sample Number : TA006  
MT Number : 10663  
Date : JUNE 23, 1986  
Description : COPPEROPOLIS, CA

Total Area Scanned : 23940.0 sq um  
Magnification : 20000 x  
Area of Original Filter : 8.55 sq cm  
Area Ashed : 4.28 sq cm  
Dilution Factor : 1.00  
Area of Membrane : 2.01 sq cm  
Volume of Air : 2.004 cubic meter  
Total number of structs : 118  
Detection Limit : 8369.4 fibers/cu meter (1 fiber)  
Reliable Detection Limit: 41847.2 fibers/cu meter (5 fibers)

\* \* BUNDLE \* \*

	Chrysotile	Amphibole	Indeterminant	Nonasbestos
Total Number of Structures Counted	51	0	4	1
Percent Total Structures	43.2	0.0	3.4	0.8
Total Structure Concentration (structures per cubic meter of air)	426841.6	0.0	33477.8	8369.4
Total Surface Area (sq um per cubic meter of air)	848651.7	0.0	64450.3	10648.8
Total Mass Concentration (ng per cubic meter of air)	210.95	0.00		
Structures > 5.0 um length Counted	5	0	0	0
Percent Structures > 5.0 um length	4.2	0.0	0.0	0.0
Struct Concentration > 5.0 um length (structures per cubic meter of air)	41847.2	0.0	0.0	0.0
Length				
geo. mean	2.2998	0.0000	1.5729	1.2000
standard deviation	2.1704	0.0000	1.2210	0.0000
Diameter				
geo. mean	0.1207	0.0000	0.1682	0.3000
standard deviation	0.1678	0.0000	0.2029	0.0000

000135



Med-Tox Associates, Inc. 1229 Morena Blvd San Diego, CA 92110 (619) 276-8843

TEM QUANTITATIVE ANALYSIS  
(AIR)

ROY F. WESTON, INC.  
120 HOWARD ST., S.420 SAN FRANCISCO, CA 415-543-5099  
GARY A. FLOYD

Job Number : 5112  
Sample Number : TA006  
MT Number : 10663  
Date : JUNE 23, 1986  
Description : COPPEROPOLIS, CA

Total Area Scanned : 23940.0 sq um  
Magnification : 20000 x  
Area of Original Filter : 8.55 sq cm  
Area Ashed : 4.28 sq cm  
Dilution Factor : 1.00  
Area of Membrane : 2.01 sq cm  
Volume of Air : 2.004 cubic meter  
Total number of structs : 118  
Detection Limit : 8369.4 fibers/cu meter (1 fiber)  
Reliable Detection Limit: 41847.2 fibers/cu meter (5 fibers)

\*\* CLUSTER \*\*

	Chrysotile	Amphibole	Indeterminant	Nonasbestos
Total Number of Structures Counted	21	0	4	1
Percent Total Structures	17.8	0.0	3.4	0.8
Total Structure Concentration (structures per cubic meter of air)	175758.3	0.0	33477.8	8369.4
Total Surface Area (sq um per cubic meter of air)	2106841.2	0.0	13825.1	5291.5
Total Mass Concentration (ng per cubic meter of air)	119815.53	0.00		
Structures > 5.0 um length Counted	6	0	0	0
Percent Structures > 5.0 um length	5.1	0.0	0.0	0.0
Struct Concentration > 5.0 um length (structures per cubic meter of air)	50216.7	0.0	0.0	0.0
Avg Length geo. mean	0.0000	0.0000	1.3774	4.0000
standard deviation	3.9804	0.0000	0.4384	0.0000
Avg Diameter geo. mean	0.2110	0.0000	0.0795	0.0500
standard deviation	0.9493	0.0000	0.0307	0.0000

000136

Med-Tox Associates, Inc. 1229 Morena Blvd San Diego, CA 92110 (619) 276-8843

PHASE CONTRAST EQUIVALENT  
(AIR)

RDY F. WESTON, INC.  
120 HOWARD ST., S.420 SAN FRANCISCO, CA 415-543-5099  
GARY A. FLOYD

Job Number : 5112  
Sample Number : TA006  
MT Number : 10663  
Date : JUNE 23, 1986  
Description : COPPEROPOLIS, CA

Total Area Scanned : 23940.0 sq um  
Magnification : 20000 x  
Area of Original Filter : 8.55 sq cm  
Area Ashed : 4.28 sq cm  
Dilution Factor : 1.00  
Area of Membrane : 2.01 sq cm  
Volume of Air : 2.004 cubic meter  
Total number of structs : 7  
Detection Limit : 0.0084 fibers/cc (1 fiber)  
Reliable Detection Limit: 0.0418 fibers/cc (5 fibers)

Structure Concentration (structures/cc)  
for  
Structures  $\geq$  5.0 um length &  
 $\geq$  0.3 um diameter

	Fiber	Bundle	Cluster	Matrix
Chrysotile	0.0000	0.0167	0.0418	0.0000
Amphibole	0.0000	0.0000	0.0000	0.0000
Indeterminant	0.0000	0.0000	0.0000	0.0000
Non-Asbestos	0.0000	0.0000	0.0000	0.0000

TOTAL ASBESTOS STRUCTURE CONCENTRATIONS:

Asbestos Structures/cc 0.0586  
Non-Asbestos Structures/cc 0.0000

000137

Med-Tox Associates, Inc. 1229 Morena Blvd San Diego, CA 92110 (619) 276-8843

TEM QUANTITATIVE ANALYSIS  
DATA LISTING  
(AIR)

ROY F. WESTON, INC.  
120 HOWARD ST., S.420 SAN FRANCISCO, CA 415-543-5099  
GARY A. FLOYD

Job Number : 5112  
Sample Number : TA006  
MT Number : 10663  
Date : JUNE 23, 1986  
Description : COPPEROPOLIS, CA

Field #	structure #	type	diameter	length	# of fibers	crossing?	ID	truelen.
1	1	bun	0.05	4.00	5	no	c	
1	2	bun	0.05	1.30	2	no	c	
1	3	bun	0.10	2.30	2	no	c	
1	4	bun	0.08	2.50	2	no	c	
1	5	fib	0.05	1.00		no	c	
1	6	fib	0.05	1.50		yes	i	3.00
1	7	clu	0.30	4.50	12	no	c	
1	8	bun	0.05	1.00	2	no	c	
1	9	fib	0.05	2.00		no	n	
1	10	bun	0.10	2.80	3	no	c	
1	11	fib	0.05	1.80		no	n	
1	12	bun	0.05	2.00	4	no	c	
1	13	fib	0.10	0.60		no	n	
1	14	fib	0.05	1.00		no	c	
1	15	clu	0.10	1.50	3	no	i	
1	16	fib	0.05	1.40		no	n	
1	17	clu	0.10	2.00	5	no	i	
1	18	bun	0.30	1.80	5	no	c	
1	19	fib	0.10	2.80		no	c	
1	20	fib	0.03	0.30		no	c	
1	21	clu	0.05	1.00	3	no	c	
1	22	fib	0.04	2.00		no	c	
1	23	fib	0.05	2.00		no	c	
1	24	bun	0.08	2.20	3	no	c	
1	25	clu	0.80	6.00	20	no	c	
1	26	fib	0.08	3.00		no	c	
1	27	fib	0.05	1.20		no	c	
1	28	clu	0.05	4.00	2	no	n	
1	29	bun	0.80	3.00	20	no	c	
1	30	bun	0.20	1.80	2	no	c	
1	31	clu	0.20	0.80	6	no	c	
1	32	fib	0.20	0.80		no	n	
1	33	fib	0.05	2.00		no	c	
1	34	clu	0.05	2.80	2	no	c	
1	35	bun	0.40	2.70	10	no	c	
1	36	bun	0.30	1.60	8	no	c	
1	37	bun	0.40	1.40	8	no	c	
1	38	fib	0.50	4.00		no	c	
2	1	clu	0.15	6.40	2	no	c	

000138



Med-Tox Associates, Inc. 1229 Morena Blvd San Diego, CA 92110 (619) 276-8843

TEM QUANTITATIVE ANALYSIS  
DATA LISTING  
(AIR)

Field #	structure #	type	diameter	length	# of fibers	crossing?	ID	truelen.
2	3	bun	0.20	2.00	2	no	i	
2	4	clu	2.00	6.60	50	no	c	
2	5	clu	0.10	1.20	4	no	i	
2	6	clu	0.04	1.00	2	no	i	
2	7	bun	0.15	1.80	4	no	c	
2	8	bun	0.05	0.80	2	no	c	
2	9	bun	0.05	4.80	2	no	c	
2	10	bun	0.05	3.00	2	no	c	
2	11	clu	3.00	7.20	50	no	c	
2	12	clu	0.50	2.00	10	no	c	
2	13	clu	0.05	1.80	3	no	c	
2	14	clu	0.20	1.00	8	no	c	
2	15	bun	0.05	0.80	2	no	c	
2	16	bun	0.08	1.20	2	no	c	
2	17	clu	0.05	0.60	2	no	c	
2	18	clu	0.05	0.30	2	no	c	
2	19	bun	0.30	4.50	10	no	c	
2	20	fib	0.05	0.60		no	c	
2	21	bun	0.08	0.60	2	no	i	
2	22	bun	0.10	6.80	3	no	c	
2	23	bun	0.10	1.60	4	no	c	
2	24	bun	0.15	3.60	4	no	c	
2	25	clu	0.08	0.00	2	no	c	
2	26	bun	0.08	0.70	2	no	c	
2	27	bun	0.10	1.20	4	no	c	
2	28	bun	0.15	1.60	3	no	c	
2	29	bun	0.20	4.00	6	no	c	
2	30	fib	0.04	2.50		no	c	
2	31	fib	0.04	1.40		no	c	
2	32	fib	0.06	2.10		no	c	
2	33	clu	0.05	1.40	2	no	c	
2	34	clu	0.30	2.00	7	no	c	
2	35	bun	0.15	2.40	3	no	c	
2	36	bun	0.08	2.80	2	no	c	
2	37	bun	0.10	1.60	3	yes	c	3.20
2	38	clu	0.05	0.80	2	no	c	
2	39	fib	0.08	3.50		no	c	
2	40	fib	0.08	1.40		no	c	
2	41	fib	0.06	1.20		no	c	
2	42	bun	0.40	2.00	20	no	c	
2	43	bun	0.50	3.40	10	no	i	
2	44	bun	0.30	1.20	3	no	n	
2	45	fib	0.05	0.70		no	c	
2	46	bun	0.05	4.80	3	no	c	
2	47	bun	0.10	0.90	2	no	c	
2	48	fib	0.05	1.20		no	c	
2	49	fib	0.06	3.00		no	c	
2	50	fib	0.08	1.00		no	c	
2	51	fib	0.05	0.80		no	c	
3	1	bun	0.10	6.70	5	no	c	

000139

Med-Tox Associates, Inc. 1229 Morena Blvd San Diego, CA 92110 (619) 276-8843

TEM QUANTITATIVE ANALYSIS  
DATA LISTING  
(AIR)

Field #	structure #	type	diameter	length	# of fibers	crossing?	ID	truelen.
3	2	bun	0.05	2.00	2	no	c	
3	3	bun	0.20	1.60	4	no	c	
3	4	fib	0.05	0.90		no	c	
3	5	bun	0.10	1.50	2	no	i	
3	6	fib	0.08	3.20		yes	c	6.40
3	7	fib	0.05	1.00		no	c	
3	8	bun	0.50	7.00	10	no	c	
3	9	bun	0.08	3.50	2	no	c	
3	10	fib	0.05	1.00		no	c	
3	11	fib	0.05	0.60		no	c	
3	12	clu	0.20	3.50	12	no	c	
3	13	bun	0.08	2.80	2	no	c	
3	14	bun	0.15	6.00	4	no	c	
3	15	bun	0.10	2.20	2	no	c	
3	16	bun	0.50	4.50	15	no	c	
3	17	bun	0.08	2.10	2	no	c	
3	18	clu	0.10	4.00	3	no	c	
3	19	bun	0.60	13.00	20	no	c	
3	20	bun	0.15	1.20	2	no	c	
3	21	bun	0.08	3.40	3	no	c	
3	22	bun	0.10	2.20	3	no	c	
3	23	bun	0.05	0.80	2	no	c	
3	24	clu	2.00	6.80	50	no	c	
3	25	clu	2.00	5.00	50	no	c	
3	26	fib	0.05	0.80		no	c	
3	27	bun	0.10	1.20	2	no	c	
3	28	fib	0.05	1.00		no	c	
3	29	fib	0.05	0.80		no	c	

000140

#### QA/QC for Asbestos Analyses

For all analyses the laboratory adheres to established EPA and NIOSH methods as the most basic foundation of quality control and to ensure comparability of data to established databases. For each analytical method further QC procedures are detailed below.

#### Phase Contrast Microscopy (PCM)

Accuracy of PCM analyses is evaluated by participation in the National Institute for Occupational Safety and Health Proficiency Analytical Testing (PAT) program. Additional accuracy checks include interlaboratory comparison of results on real samples and intralaboratory comparison of results between different analysts.

Precision of PCM analyses is determined by blind replicate analyses on 10% of samples.

Blank levels are determined on field blanks and lab blanks which number 10% of the total samples.

#### Polarized Light Microscopy (PLM)

Accuracy of PLM analyses is evaluated by participation in the Environmental Protection Agency Bulk Asbestos Quality Assurance Program. Additional checks on accuracy include interlaboratory comparison of results on real samples and intralaboratory comparisons between different analysts. Our lab also maintains a reference set of bulk asbestos samples including UICC asbestos standards and samples collected from various U. S. mines by our staff. Our bulk standards have been analyzed by benchmark mineralogical techniques including electron microscopy and x-ray diffraction and are superior to standards available commercially.

Precision of PLM analyses is determined by blind replicate analyses on 10% of samples.

Contamination of bulk samples is controlled by ensuring that only qualified personnel collect the samples in approved containers.

#### Transmission Electron Microscopy (TEM)

Accuracy of TEM analyses is evaluated by analysis of National Bureau of Standards, Standard Reference Material #1876 (asbestos fibers on air filters). Additional checks on accuracy include spiking filters with known aqueous asbestos standards to check asbestos mass concentrations and to verify proper identification of asbestos mineral species. Interlaboratory and intralaboratory analytical comparisons on real samples also play a fundamental part in our QC program.

Precision of TEM analyses is determined by blind replicate analyses on 5% of samples.

Blank levels are determined on field blanks and lab blanks which number 10% of the total samples.

000140.001





## OCCUPATIONAL & ENVIRONMENTAL HEALTH SERVICES

1229 Moreno Blvd • San Diego, California 92110 • (619) 276-8843

### FORMULA FOR CALCULATING AIR VOLUMES REQUIRED DURING ASBESTOS MONITORING

$$\frac{f}{F} \times \frac{A}{a} \times \frac{1}{1000} \times \frac{1}{C} = V$$

- f** = minimum number of fibers to achieve a statistically significant detection limit, e.g. 10 fibers for PCM and 5 fibers for TEM
- F** = number of fields counted, e.g. 100 fields for PCM and 10 fields for TEM
- A** = effective collection area of original filter, e.g. 385 mm<sup>2</sup> for 25 mm diameter filter and 855 mm<sup>2</sup> for 37 mm diameter filters
- a** = area of microscopic field of view, e.g. 0.00785 mm<sup>2</sup> for a properly calibrated Walton-Beckett graticule (PCM) and 0.0072 mm<sup>2</sup> for a TEM grid square
- C** = fiber concentration of desired detection limit, e.g. 0.01 fibers/cm<sup>3</sup>
- V** = minimum air volume which must be sampled to achieve desired detection limit (result in liters if above units are used)

#### Worked Examples:

for PCM  $\frac{10}{100} \times \frac{385}{0.00785} \times \frac{1}{1000} \times \frac{1}{.01} = 490 \text{ liters}$

for TEM  $\frac{5}{10} \times \frac{385}{0.0072} \times \frac{1}{1000} \times \frac{1}{.01} = 2674 \text{ liters}$

If you have any questions concerning variables in the above equation, please feel free to call me.

Tom Dagenhart  
San Diego Lab Manager

BRANCH OFFICES  
PLEASANT HILL

000141

SEATTLE

TUSTIN